

APPLICATION OF SULFUR  
DYES ON COTTON AT  
HIGH TEMPERATURE

WILLIAM ARTHUR MURAUSKAS

Thesis  
M96

Library  
The College of Graduate School  
Berkeley, California











4157  
127

**APPLICATION OF SULFUR DYES  
ON COTTON  
AT HIGH TEMPERATURE**

**A THESIS**

**Presented to  
the Faculty of the Graduate Division  
Georgia Institute of Technology**

**In Partial Fulfillment  
of the Requirements for the Degree  
Master of Science in Textile Engineering**

**By  
William Arthur Murauskas  
June 1954**

Map

THE UNITED STATES OF AMERICA

DEPARTMENT OF THE INTERIOR

BUREAU OF LAND MANAGEMENT

1916

SECTION 1

OF THE

LANDS OF THE UNITED STATES

IN THE STATE OF CALIFORNIA

SECTION 2

OF THE

LANDS OF THE UNITED STATES

1916

SECTION 3

OF THE



Library  
The University of Tennessee Graduate School  
Knoxville, Tennessee

**APPLICATION OF SULFUR DYES  
ON COTTON  
AT HIGH TEMPERATURE**



### ACKNOWLEDGMENTS

In due recognition of the assistance received which contributed to the successful completion of this thesis, a sincere expression of gratitude is offered to the United States Naval Postgraduate School, under the auspices of which this course of study was undertaken; to Dr. James L. Taylor, of the Georgia Institute of Technology Textile School, who actively directed and guided the work performed in this thesis; and to the personnel of the Atlanta Research Laboratory of the E. I. duPont de Nemours and Company, Inc., who provided advice and materials used in the laboratory research phase of this investigation.



## TABLE OF CONTENTS

	Page
ACKNOWLEDGMENTS . . . . .	ii
LIST OF TABLES . . . . .	iv
LIST OF ILLUSTRATIONS . . . . .	vi
SUMMARY . . . . .	vii
 CHAPTER	
I. INTRODUCTION . . . . .	1
The Sulfur Dye	
The High Temperature Theory	
The Problem	
Literature Survey	
II. INSTRUMENTATION AND EQUIPMENT . . . . .	14
III. PROCEDURE . . . . .	19
Characterization of the Yarn	
Scouring	
Sample Preparation	
Colorimetry	
Dyeing	
Dyed Yarn Strength	
Light-Fastness Tests	
Wash-Fastness Tests	
IV. DISCUSSION OF RESULTS . . . . .	32
V. CONCLUSIONS . . . . .	42
VI. RECOMMENDATIONS . . . . .	44
APPENDIX . . . . .	45
BIBLIOGRAPHY . . . . .	75

# CONTENTS OF VOLUME

1990

11	.....	1990-1991
12	.....	1991-1992
13	.....	1992-1993
14	.....	1993-1994

.....

15	.....	1994-1995
----	-------	-----------

16	.....	1995-1996
----	-------	-----------

17	.....	1996-1997
----	-------	-----------

18	.....	1997-1998
----	-------	-----------

19	.....	1998-1999
----	-------	-----------

20	.....	1999-2000
----	-------	-----------

21	.....	2000-2001
----	-------	-----------

22	.....	2001-2002
----	-------	-----------

23	.....	2002-2003
----	-------	-----------

24	.....	2003-2004
----	-------	-----------



## LIST OF TABLES

Table	Page
1. List of Dyes Used . . . . .	33
2. Dyeing Cycle Analysis of Sulfogene Carbon HCF Grains . . . . .	34
3. Comparison of Dyebath Exhaustions for Sulfogene Carbon HCF Grains . . . . .	35
4. Comparison of Dyebath Exhaustions for Sulfogene Navy Blue 4RCF Supra . . . . .	36
5. Comparison of Dyebath Exhaustions for Sulfogene Tan 2RCF . . . . .	37
6. Comparison of Dyebath Exhaustions for Sulfogene Direct Blue BRCF Conc. 200% . . . . .	38
7. Comparison of Dyebath Exhaustions for Sulfogene Brilliant Green GCF Extra Conc. 150% . . . . .	39
8. Yarn Number of Scoured, Undyed Cotton Yarn . . . . .	46
9. Twist of Scoured, Undyed Cotton Yarn . . . . .	47
10. Single Strand Breaking Strength of Scoured, Undyed Cotton Yarn . . . . .	48
11. Light Absorption Percentages for Sulfogene Carbon HCF Grains . . . . .	49
12. Light Absorption Percentages for Sulfogene Navy Blue 4RCF Supra . . . . .	50
13. Light Absorption Percentages for Sulfogene Tan 2RCF . . . . .	51
14. Light Absorption Percentages for Sulfogene Direct Blue BRCF Conc. 200% . . . . .	52
15. Light Absorption Percentages for Sulfogene Brilliant Green GCF Extra Conc. 150% . . . . .	53





Table	Page
16. Light Transmission Percentages for Sulfogene Carbon HCF Grains . . . . .	54
17. Light Transmission Percentages for Sulfogene Navy Blue 4RCF Supra . . . . .	56
18. Light Transmission Percentages for Sulfogene Tan 2RCF . . . . .	58
19. Light Transmission Percentages for Sulfogene Direct Blue BRCF Conc. 200% . . . . .	60
20. Light Transmission Percentages for Sulfogene Brilliant Green GCF Extra Conc. 200% . . . . .	62
21. Dyebath Exhaustion for Sulfogene Carbon HCF Grains at 190° F. for Sixty Minutes . . . . .	64
22. Dyebath Exhaustion for Sulfogene Carbon HCF Grains at High Temperature . . . . .	65
23. Dyebath Exhaustion for Sulfogene Navy Blue 4RCF Supra at 190° F. for Sixty Minutes . . . . .	66
24. Dyebath Exhaustion for Sulfogene Navy Blue 4RCF Supra at High Temperature . . . . .	67
25. Dyebath Exhaustion for Sulfogene Tan 2RCF at 190° F. for Sixty Minutes . . . . .	68
26. Dyebath Exhaustion for Sulfogene Tan 2RCF at High Temperature . . . . .	69
27. Dyebath Exhaustion for Sulfogene Direct Blue BRCF Conc. 200% at 190° F. for Sixty Minutes . . . . .	70
28. Dyebath Exhaustion for Sulfogene Direct Blue BRCF Conc. 200% at High Temperature . . . . .	71
29. Dyebath Exhaustion for Sulfogene Brilliant Green GCF Extra Conc. 150% at 190° F. for Sixty Minutes . . . . .	72
30. Dyebath Exhaustion for Sulfogene Brilliant Green GCF Extra Conc. 150% at High Temperature . . . . .	73
31. Comparison of Residual Strengths of Dyed Yarns . . . . .	74

Page	Page
101	101
102	102
103	103
104	104
105	105
106	106
107	107
108	108
109	109
110	110
111	111
112	112
113	113
114	114
115	115
116	116
117	117
118	118
119	119
120	120
121	121
122	122
123	123
124	124
125	125
126	126
127	127
128	128
129	129
130	130
131	131
132	132
133	133
134	134
135	135
136	136
137	137
138	138
139	139
140	140
141	141
142	142
143	143
144	144
145	145
146	146
147	147
148	148
149	149
150	150

## LIST OF ILLUSTRATIONS

Figure	Page
1. Morton One-Pound Package Machine (Modified), Top View . . . . .	16
2. Morton One-Pound Package Machine (Modified), Side View . . . . .	17
3. Lunetron Colorimeter, Model 402-E . . . . .	18
4. Sulfogene Carbon HCF Grains Graph of Concentration vs. Percentage Light Transmission . . . . .	55
5. Sulfogene Navy Blue 4RCF Supra Graph of Concentration vs. Percentage Light Transmission . . . . .	57
6. Sulfogene Tan 2RCF Graph of Concentration vs. Percentage Light Transmission . . . . .	59
7. Sulfogene Direct Blue 8RCF Conc. 200% Graph of Concentration vs. Percentage Light Transmission . . . . .	61
8. Sulfogene Brilliant Green GCF Extra Conc. 150% Graph of Concentration vs. Percentage Light Transmission . . . . .	63





APPLICATION OF SULFUR DYES  
ON COTTON  
AT HIGH TEMPERATURE

SUMMARY

The purpose of this investigation was twofold. The first, and more general, aim was to contribute, in some small measure, to the knowledge of dyeing by determining what benefits, if any, can be realized from the high temperature technique in applying sulfur dyes to cotton. In this instance, it is considered that even negative information will be of considerable value if only by virtue of the fact that the question must be answered if the field of high temperature is to be explored thoroughly. The second, and more specific, aim was to determine whether industry can utilize profitably the high temperature method in applying sulfur dyes to cotton with no more special equipment than the pressure-closed dyeing systems that it already has at its disposal. In this instance, only positive information is of value.

During the dyeing phase of this work, dye-house-like conditions were maintained in preference to laboratory conditions to satisfy the above-mentioned second aim. The dyeings were performed in a Horton one-pound package machine,





modified to permit utilization of the entire dyebath at the high temperature rather than only about half the dyebath, as was the case prior to modification.

Five sulfur dyes, representative of the ranges of light-fastness and wash-fastness available to this class, were selected for use in the problem. Four one-pound packages of cotton yarn were dyed with each color; two runs being made at a normal temperature of 190° F.; and two runs being made at a high temperature of 250° F., under sufficiently controlled conditions to provide a basis for comparison. A preliminary high temperature run was made to determine the optimum duration of the high temperature phase of the dyeing. All subsequent high temperature runs were conducted in accordance with this determination. The normal temperature runs were conducted, insofar as was practicable, in accordance with the procedures recommended by the manufacturer of the dyestuffs.

The results of the two techniques were compared, for each dye, on the following bases:

- (1) the duration of the dyeing cycle
- (2) the exhaustion of the dyebath
- (3) light-fastness
- (4) wash-fastness
- (5) the residual strength of the dyed yarn.

In every case, the high temperature technique proved superior to the normal temperature method both in the



duration of dyeing cycle and in exhaustion of the dyebath. No significant sacrifice of light-fastness, wash-fastness, or yarn strength was experienced.

The most valued finding of this thesis is the fact that certainly equal, and, in many cases, better, dyeing results can be obtained through the use of high temperature techniques at a thirty-three per cent saving in time.

The wide range of variation between the performances of the individual dyes points to the possibility that some members of this class may not be applied profitably at a high temperature. Extensive research efforts on the part of individuals throughout the world have established the value of the high temperature technique sufficiently well to warrant wide-scope research on the part of the dyestuff manufacturers into the adaptability of each of their dyes to application at a high temperature. It is suggested that such information be put at the disposal of the dyeing industry, so that those who are able to could avail themselves of the economies and other benefits to be realized from the use of high temperatures.





## CHAPTER I

### INTRODUCTION

The year 1873 marked the inception of sulfur dyes when two inspired Frenchmen, Croissant and Bretonniere, giving full reign to their imaginations and hopes, melted sulfur and sodium sulfide together with sawdust, blood, peat, and other "organic substances" to come up with a successful coloring matter which they proceeded to name "Cachou de Laval". The connection with 'Laval' is not known, but the word, 'Cachou', goes back to the Malayan word, 'catechu', which refers to various products of particular tropical plants which were used for dyeing, tanning, and even for preserving fish nets. In view of the foregoing, it is easy to understand the inability of either Croissant or Bretonniere to produce a satisfactory description of the exact chemical constitution of their end product. Unfortunately, this stigma of uncertainty has plagued the sulfur dye throughout its history, even to the present day.

It is only fair to say, however, that a good deal of the mystery has been cleared away as far as the modern sulfur dye is concerned. The work started only twenty years after Croissant and Bretonniere's experiment when, in 1893, Vidal,

1

THE

PROLOGUE

THEY WERE IN THE MIDDLE OF THE ROAD, AND

THEY WERE IN THE MIDDLE OF THE ROAD, AND

THEY WERE IN THE MIDDLE OF THE ROAD, AND

THEY WERE IN THE MIDDLE OF THE ROAD, AND

THEY WERE IN THE MIDDLE OF THE ROAD, AND

THEY WERE IN THE MIDDLE OF THE ROAD, AND

THEY WERE IN THE MIDDLE OF THE ROAD, AND

THEY WERE IN THE MIDDLE OF THE ROAD, AND

THEY WERE IN THE MIDDLE OF THE ROAD, AND

THEY WERE IN THE MIDDLE OF THE ROAD, AND

THEY WERE IN THE MIDDLE OF THE ROAD, AND



another Frenchman, melted sulfur and sodium sulfide together with intermediates of known chemical constitution, para aminophenol and dinitrophenol, to produce a black sulfur dye of immediate success, appropriately named, "Vidal's Black". Using such intermediates of known chemical constitution, of course, equips the experienced chemist with considerably more information on which to base an opinion as to the constitution of the end product. Work has progressed at length to the point where, in current times, the chemical constitution, and even the structure, of the building units of sulfur dyes is known. But the arrangement of these units in the dye structure and the exact number that go to make up a complete molecule are still matters which have not been determined with requisite scientific accuracy.

The entire difficulty in the matter stems from the fact that sulfur dyes, at best, are mixtures of products of the original members of the reaction. The sulfur dyestuff, itself, which would be the chief coloring agent in the mixture, is extremely difficult to isolate. As it approaches purity, it becomes less and less soluble, even in the alkali sulfide solutions normally used to dissolve the dyes.

The sulfur dye's indifference to chemical analysis, however, has not detracted from its long-standing success and popularity. Though the full range of colors is not available in the sulfur dyes, and though the colors that are available produce far from brilliant shades, the cheapness of the dye





and its generally good fastness to light, washing, and crocking have made it a natural choice for heavy cotton fabrics, industrial fabrics, and work clothing. For years, the dye-stuff industry has produced more sulfur blacks than any other dye, and, to this day, they stand among the top three or four dyes in production.

In recent years, sulfur dyes have lost a share of their original popularity to newer, or improved, competitive products. However, the development of a dyeing technique which would improve upon the efficiency of present methods could do much to bolster the status of what is, essentially, a good dye handicapped with poor exhaustion properties.

The most important recent development in the field of dyeing textiles, the high temperature technique, could offer some promise for this class of dyes. It has already led to signal success in applying colors to the "hard-to-dye" synthetics, and it greatly speeds the dyeing of wool (1). The whole science of dyeing is based almost solely on empirical knowledge. If all the benefits to be gained from the use of the high temperature technique are to be realized, then the fund of the empirical knowledge of dyeing must be enlarged to include the various effects of high temperatures on all dyes and fibers in their usual combinations. This work is intended to supply some of that information for the sulfur dyes and the cotton combination.

High temperatures work to advantage in many dyeing operations through various mechanisms, all stemming from the





thermal energy increase of the fiber-dyebath system. For the fiber, the additional heat aids in swelling the mass, thus serving to enlarge the macro-molecular interstices. This, of course, provides easier entry for the dye molecule, or aggregate, into the interior of the fiber.

As for the dyes, very few are known to go into complete solution at room temperature in the usual dyebath concentrations. Instead, they tend to form aggregates of anywhere up to one hundred molecules, depending upon the nature of the particular dyestuff under consideration. This works to no advantage in the dyeing operation. The dye molecules, themselves, are characteristically large in structure, often approaching the interstices of the fiber in size. Common aggregations of these massive molecules can, and often do, exceed the size of the interstices. Good dyeing cannot be realized if the dye particles are too large to enter into the interior of the fiber.

However, it is also known that an increase in the temperature or an extreme dilution of the solution will act to break down these aggregates. Since a very dilute solution does not serve as a good working dyebath, the only practical way to reduce the degree of aggregation of the dye is to raise the temperature of the bath. Some dyes will disperse satisfactorily at, or below, the boiling temperature. Others will not. Most dispersions can be improved by raising the temperature above the boil.

the same time, the fact that the government has been able to  
 maintain a high level of employment in the public sector  
 has been a major factor in the success of the government's  
 economic policy. The fact that the government has been able to  
 maintain a high level of employment in the public sector  
 has been a major factor in the success of the government's  
 economic policy.

It is true that the government has been able to maintain a  
 high level of employment in the public sector, but it is also  
 true that the government has been able to maintain a high  
 level of employment in the private sector. The fact that the  
 government has been able to maintain a high level of  
 employment in the public sector has been a major factor in  
 the success of the government's economic policy. The fact  
 that the government has been able to maintain a high level  
 of employment in the private sector has been a major factor  
 in the success of the government's economic policy. The fact  
 that the government has been able to maintain a high level  
 of employment in the public sector has been a major factor  
 in the success of the government's economic policy. The fact  
 that the government has been able to maintain a high level  
 of employment in the private sector has been a major factor  
 in the success of the government's economic policy.

It is true that the government has been able to maintain a  
 high level of employment in the public sector, but it is also  
 true that the government has been able to maintain a high  
 level of employment in the private sector. The fact that the  
 government has been able to maintain a high level of  
 employment in the public sector has been a major factor in  
 the success of the government's economic policy. The fact  
 that the government has been able to maintain a high level  
 of employment in the private sector has been a major factor  
 in the success of the government's economic policy. The fact  
 that the government has been able to maintain a high level  
 of employment in the public sector has been a major factor  
 in the success of the government's economic policy. The fact  
 that the government has been able to maintain a high level  
 of employment in the private sector has been a major factor  
 in the success of the government's economic policy.

High temperature also increases the migratory activity of the dye particles. This causes earlier and better penetration of the fiber by the dye. It also encourages better levelling of the deposited color. A dye, by its own definition, must have a characteristic affinity for its fiber. It derives its affinity from hydrogen bonding or residual valences. Rarely do a dye and a fiber combine chemically in the common sense of the word. Under normal conditions, remembering that they never lose their tendency to aggregate, the dye particles often accumulate on selected areas of the goods at the expense of more lightly laden areas. Under higher temperatures, this tendency merely to aggregate on the fiber is counteracted by the higher kinetic energy generated by the heat. The dye particles which would be in excess in any one area receive sufficient motive force to migrate to a more sparsely laden area where the opportunity for permanent bonding with the fiber is much greater. The end result is better levelling of the color.

The problem at hand was to determine whether these high temperature principles can be applied profitably to the dyeing of cotton with sulfur dyes. To be profitable, the operation must save either time or material.

Normally, sulfur dyes do not exhaust well. The cotton preferentially absorbs only twenty to thirty per cent of the dyestuff from the bath. For economy's sake, the one bath must be used for several runs, with periodic replenishment



The first thing I noticed when I stepped out of the car was the cold. It was a sharp contrast to the warm blanket of the car. I shivered as I walked towards the entrance of the building. The air was thick with the scent of old books and the sound of footsteps on the polished floor. I felt a sense of anticipation as I approached the door. The door was slightly ajar, and I pushed it open. The interior was dimly lit, with the light coming from the windows. I saw a man in a suit standing behind a desk, looking at some papers. He looked up at me as I entered. I gave him a slight nod and walked towards the back of the room. I found a small, empty room with a desk and a chair. I sat down and looked at the clock on the wall. It was 10:15. I took a deep breath and looked at the door. I knew I had to go in. I stood up and walked towards the door. I opened the door and stepped out. The air was cold, but I felt a sense of relief. I walked towards the car and got in. I started the engine and drove away. The car was warm and comfortable. I felt a sense of peace as I drove. I knew I had made it. I had survived. I had won. I had won.



of the exhausted dye increment. This cannot be continued indefinitely, though. The dye does age on standing, and will lose some of its coloring strength as a consequence. Eventually, the standing bath must be replaced with an entirely fresh one. If the exhaustion of the dyebath could be increased, then, literally, less material and money would go down the drain.

Even more important than the saving of material, since sulfur dyes are relatively cheap, is the saving of time. Time, in a profit and loss statement of operation, becomes translated into terms of labor and effective employment of equipment. Both items are commonly among the most expensive in most enterprises. If three high temperature dyeing runs could be completed in the time that it normally takes to do two normal temperature runs, then, in effect, the production capacity of the plant would be increased automatically by fifty per cent without the addition of a single piece of equipment.

All savings are to no avail, however, if the high temperature product is not as good as the normal temperature product. The best gages for comparison are fastness to light and washing, and residual strength of the yarn. None should suffer damage from the higher temperature. Or, at least, no damage should be so extensive as to offset any benefits to be gained.





Unfortunately, we are illy equipped to engage in theoretical conjecture on this matter. Though we are learning fast, too little is now known about the physico-chemical properties of dyes and their fibers. The enigma of the sulfur dye's chemical structure makes the problem even more complex in this case. Venkataraman (2) nicely implies the futility of any attempt at prediction when he states,

On the theoretical side, curiously enough, color chemistry is almost a virgin field. Our knowledge of the relation between the chemical constitution of dyes and their color is largely empirical. The physicists have not yet been able to proceed beyond a few hydrocarbons in their application of quantum mechanical methods to the interpretation and prediction of absorption spectra. Little is known of the precise action of light on dyes when fading takes place, and of any relation between the color and the chemical constitution of a dye on the one hand and its fastness to light on the other. Concerning the catalytic activity of certain dyes in the photo-chemical degradation of cellulose, several problems remain unsolved. The theories of dyeing are in an extremely rudimentary condition, notwithstanding the spate of publications on the subject from time to time.

Moreover, the sulfur dyes are a family only by virtue of their common method of preparation, that is, the fusing together of various organic intermediates with sulfur, sodium sulfide, and/or polysulfides (3). It is not reasonable to expect that any one member will represent the behavior of the entire family.

In view of the foregoing, only one course of action remained open. The empirical method was the only attack which could yield sufficiently concrete answers for the solution of this problem. Any theorizing was beyond its scope





and will be left for the time when our knowledge of sulfur dyes and the chemistry of dyeing is less sketchy than it is at present.

The most thorough treatment of the theory of high temperature dyeing is that done by Drijvers (4), and his will be the guiding principles in the handling of this problem. The following is a review of those principles.

Dyeing can be considered to be a physico-chemical, or a purely chemical, reaction. This reaction is reversible, and will reach an equilibrium eventually. The state of the equilibrium and the time within which it is established are functions of the temperature under which the reaction is carried out.

There are three phases involved in the dyeing cycle. The first is the partial diffusion of the dyestuff in the bath toward the surface of the fiber. The dye molecules, by virtue of their residual valences, also tend to aggregate together in the dyebath. The degree of aggregation differs from one dyestuff to another. For each temperature, there is a corresponding state of equilibrium between the quantity of dye in the molecular state and the quantity of dye in the aggregated state.

The second phase is the adsorption of the dye by the fiber. The molecularly diffused dye is brought into contact with the fiber by the circulating bath. The adsorption



phenomenon proceeds up to the point of the equilibrium that corresponds to the particular temperature prevailing.

The third phase is the diffusion of the dye into the interior of the fiber. The dye molecule must penetrate the intercrystalline spaces of the cellulose macro-molecule. Penetration, normally, is slow and difficult because of the mechanical resistance which the dye meets in progressing through the fiber. Consequently, the dye piles up and rapidly accumulates on the surface of the fiber, hastening the state of equilibrium. As a result, adsorption stops, but diffusion into the interior of the fiber is encouraged. High temperature greatly aids in the penetration process. The addition of electrolytes does encourage an early arrival at the state of physico-chemical equilibrium, but temperature still remains as the primary determinant.

A dynamic system of a dyebath and a mass to be dyed cannot be driven toward practical exhaustion until a tinctorial equilibrium has been reached between the entire mass and the whole of the dyebath. This consideration is of prime importance to level dyeing. The principal factors having the power to influence this system are temperature and concentration of electrolyte. The temperature most importantly influences the speed with which dynamic equilibrium is reached. The manner by which equilibrium is reached, the quantity of dyestuff, and the amount of material to be dyed in its presence at the moment of equilibrium are characteristics belonging to each individual dye.







Practical application of these principles involves raising the temperature as rapidly as possible to the point most favorable to the speed of diffusion of the dye and to the early establishment of the physico-chemical equilibrium. This promotes good penetration and levelling. Then the bath is cooled to the point most favorable to exhaustion. The addition of electrolyte will increase this value. In essence, the technique is the reverse of the normal order of dyeing.

Perhaps the most ambitious undertaking in high temperature research was the work done by the Philadelphia Section of the American Association of Textile Chemists and Colourists (5). Their announced purpose was to investigate, briefly, the behavior of every class of dye and all the common fibers under high temperature. Their report is the only one that carries any information on sulfur dyes at high temperatures. They worked with five dyes of this class and found that a temperature of 236° F. would destroy up to five per cent of the coloring power, though in one case (Sulfogene Golden Brown ECF) improvement of five per cent was shown. They offer the opinion that the cotton fiber should be able to withstand the same temperatures and conditions of alkalinity in sulfur dyeing that it does in kier boiling.

In their actual dyeing runs, they padded cotton fabric with a solution of thirty grams per liter of dye solubilized in an equal amount of sodium sulfide. This they dried and then treated in a glass pressure tube with a one-to-ten

...and the ... of the ...

...the ... of the ...

...the ... of the ...

...the ... of the ...

...the ... of the ...

...the ... of the ...

...the ... of the ...

...the ... of the ...



fabric-to-bath ratio of seven and one-half grams per liter of sodium sulfide at a temperature of 236° F. for thirty minutes. They judged their results by a visual rating of the tinctorial strength and shade, and found an increase in strength in every case but one which did, at least, equal the results of the normal temperature run of 180° F.

The high temperature produced no change in the dyes' original fastness ratings.

They did not attempt to measure and compare bath exhaustions.

Their findings indicated that, though the tinctorial strengths of the sulfur dyeings at high temperature increased only slightly, good dyeings might be obtained by much shorter periods of exposure to the high temperature.

Among their general conclusions, they state that high temperatures greatly increase diffusion and penetration, and that speed, levelling, and diffusion gains make high temperatures worthy of adaption to high-speed, continuous dyeing processes where volume production can make the most of the economies to be gained.

Zimmerman (1), though not referring directly to sulfur dyes, does list some of the more important general aspects of high temperature dyeing.

He reports that high temperature dyeing is fifteen years old. Pressure, itself, has no effect on the process, but is used merely to arrive at temperatures above the boil.





The rates of most chemical reactions increase with temperature, and the rate of dyeing similarly increases. However, some dyes decompose when held at a high temperature for several hours. Most machines dye at 250° F. and forty pounds gage pressure. Little advantage is gained by the use of a higher temperature.

Dyeing takes place by the diffusion of dye molecules into the intermicellar spaces of the fiber. Fiber swells in water. Wool, in particular, undergoes greatly increased swelling as the temperature is increased. At the same time, the rate of diffusion of the dye particles increases with temperature and with a decrease in their size. Few dyes are known to go into molecular dispersion in aqueous solutions. They commonly aggregate into groups of from two to ten molecules, sometimes as many as a hundred molecules. The degree of aggregation varies inversely with the temperature.

In summation, high temperature promotes dyeing by increasing the pore sizes in the fiber; by decreasing the size of the dye particles; and by increasing the rate of diffusion of the dye. Cellulosic materials do not respond to high temperatures as well as do protein materials. Also, under equal conditions, a fiber at equilibrium takes up less dye at higher temperatures, but the rate of diffusion of the dye is increased. In general, high temperature dyeing takes less time and produces equal or better fastness ratings in comparison with normal temperatures.



As for damage to textile fibers, none suffer significantly from exposure to high temperatures for a short period of time, with the exception of cellulose acetate.

With the exception of Drijvers' work, it appears that practically all high temperature research has been done using laboratory techniques and highly specialized equipment such as mercury column seals, pressure-differential platform valves, and high-pressure glass capsules. None of these hold great promise for economical reproduction on an industrial scale. Further, no reports have been found dealing with the specific aspects of high temperature application of sulfur dyes under simulated dye-house conditions. It is felt that this work can help to bridge the gap between laboratory and dye-house by attempting to provide answers which will reflect directly upon considerations of immediate practical application of sulfur dyes at high temperatures on a commercial basis.







## CHAPTER II

### INSTRUMENTATION AND EQUIPMENT

The following list comprises all the items of special equipment used in this problem:

Morton Twelve-Pound Package Machine

Morton One-Pound Package Machine (Modified)

Atlas Fade-Ometer

Atlas Launder-Ometer

Lunetron Colorimeter, Model 402-E

With the exception of the modified Morton One-Pound Package Machine, all items of equipment are standard manufacturer's products, available on the open market, and require no special descriptive treatment.

The Morton One-Pound Package Machine was modified by the addition of a stainless steel cover which can be bolted to the top of the expansion chamber. This seals the chamber from the atmosphere and permits the entire dyebath to be circulated under high temperature.

Before modification, the expansion chamber had to be closed off from the remainder of the system by means of valves whenever it was necessary to use high temperatures. Any dye solution remaining in the expansion chamber, of course, could not be used during the high temperature phase of the dyeing. This resulted in the loss of all control over

# THE END

## THE END OF THE WORLD

The following text is a collection of words and phrases that are arranged in a specific order. The words are: "The", "end", "of", "the", "world", "is", "near", "and", "we", "are", "all", "going", "to", "die". The phrases are: "The end of the world is near", "We are all going to die", "The world is going to end", "We are all going to die", "The world is going to end", "We are all going to die". The words and phrases are arranged in a specific order that is not necessarily logical or meaningful. The words are: "The", "end", "of", "the", "world", "is", "near", "and", "we", "are", "all", "going", "to", "die". The phrases are: "The end of the world is near", "We are all going to die", "The world is going to end", "We are all going to die", "The world is going to end", "We are all going to die".

dye-bath ratios. Two views of the modified machine are shown in Figures 1 and 2.

A view of the Lunatron Colorimeter, Model 402-E, is shown in Figure 3.

All other equipment are standard items commonly found in any laboratory connected with chemistry and dyeing.

the subject of the present report, the subject of the present report.

It is the purpose of the present report to

show that the present report is the subject of the present report.

It is the purpose of the present report to

show that the present report is the subject of the present report.

It is the purpose of the present report to



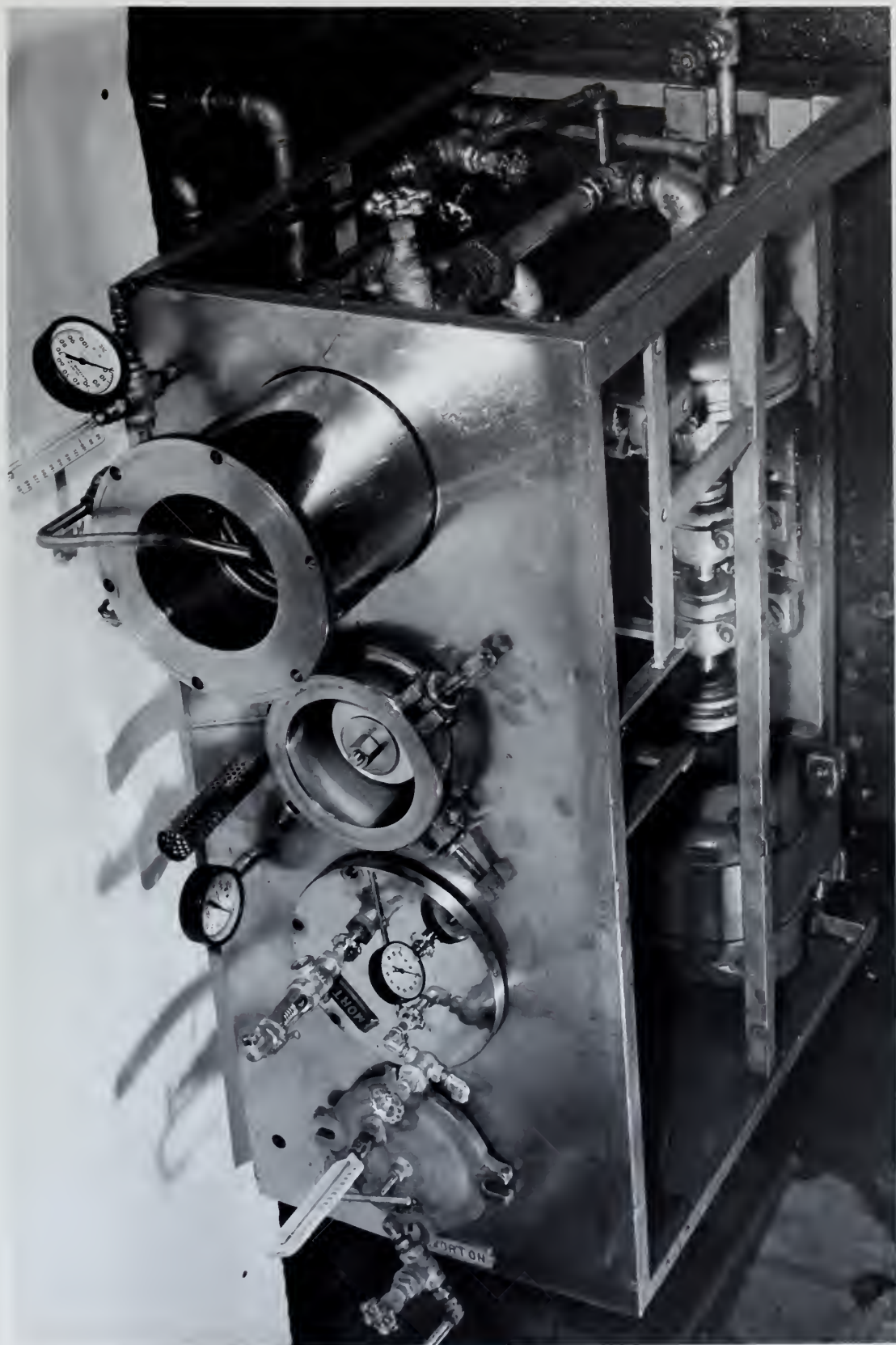


Figure 1. Morton One-Pound Package Machine (Modified)  
Top View



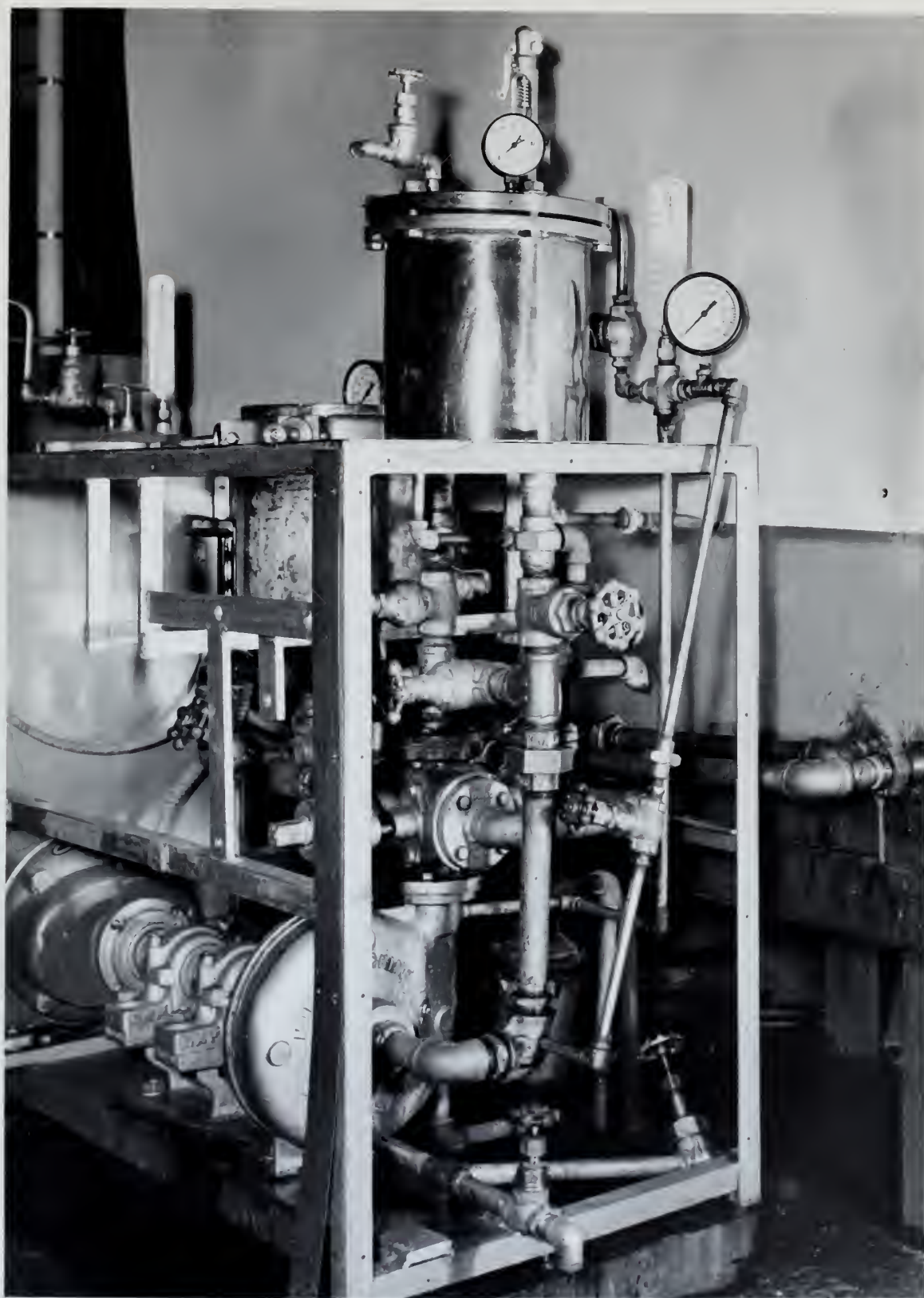


Figure 2. Morton One-Pound Package Machine  
(Modified), Side View







Figure 3. Lumetron Colorimeter, Model 402-E



## CHAPTER III

### PROCEDURE

Characterization of the Yarn.--The cotton yarn to be used in this problem was first characterized with regard to size, twist, and breaking strength in accordance with the American Society for Testing Materials Standard Test D 180-52T (6). It was found to be a number 16 single-ply yarn with a 2 twist of 18.1 turns per inch and with a breaking strength of 1.15 pounds. The strength, however, was found to be highly irregular, varying from 0.9 to 1.62 pounds. This irregularity is expected to affect the accuracy of the strength tests on the dyed samples. The results are shown in Tables 8, 9, and 10.

After characterization, the yarn was wound on perforated, stainless-steel dyeing tubes to yield around one pound, net, per package.

Scouring.--Rather than scour each package of yarn singly in the one-pound machine just prior to dyeing, the yarn was scoured in groups in the twelve-pound machine, thus saving time in the subsequent dyeing operations.

In scouring, the yarn was first wet out with one-half per cent of Tergitol Penetrant 7 at 180° F. for fifteen minutes. The bath was set, then, at 200° F., and the actual





scouring was done in five per cent sodium hydroxide, three per cent sodium carbonate, and one per cent soap flakes for one hour. All percentages are based on the net weight of the yarn. The bath was drained, and alternate hot and cold rinses were applied until red litmus paper no longer registered alkalinity in the bath. The packages were hydro-extracted and then dried in a hot air oven for thirty-six hours. This was followed by a forty-eight hour conditioning at sixty-five per cent relative humidity and 70° F.

Sample Preparation.—Each package of scoured yarn was tagged with a number which it would carry throughout the experiment.

Eight breaking strength tests were made on each of ten packages, five of which were to be dyed at normal temperature (one for each dye used), and five to be dyed similarly at high temperature. The strength data were recorded by package number to serve as the basis for determining the comparative effects on yarn strength of each of the dyes both under normal temperature and under high temperature applications.

These data are meant to serve merely in the capacity of a control and not as an exhaustive test, inasmuch as all published literature indicates that cotton is not expected to suffer significantly from exposure to temperatures of the order to be used in this experiment (1), (5), and (7).

The weight of each package was determined to within one-tenth of a gram. This information was recorded by



package number to serve as the basis for the calculation of all weights of dyes and chemicals to be used in the processing of the packages.

Colorimetry.—Colorimetric measurements were used to determine the exhaustion of the dyebath.

Colorimetry is based on two laws (8). The first, known as Lambert's law, states that the amount of monochromatic light transmitted by a pure, colored solution is a function of the thickness of the layer of the solution through which the light is transmitted. This is expressed mathematically as,

$$I_t = I_0 \cdot e^{-al} \quad (1)$$

where  $I_t$  is the intensity of the light transmitted through the solution,  $I_0$  is the intensity of the source light being directed into the solution,  $l$  is the thickness of the layer of solution, and  $a$  is a constant.

The second law, known as Beer's law, states that the amount of monochromatic light transmitted by a pure, colored solution is a function of the concentration of the solute, in molecular dispersion, in the solution. This is expressed mathematically as,

$$I_t = I_0 \cdot e^{-bc} \quad (2)$$

where  $c$  is the concentration of the solution,  $b$  is a constant, and the remaining symbols are the same as those named above.



the following conditions are satisfied: (i) the function  $f$  is continuous on  $[a, b]$ ; (ii) the function  $f$  is differentiable on  $(a, b)$ ; (iii) the function  $f$  is not constant on  $[a, b]$ . Then the function  $f$  is strictly increasing on  $[a, b]$ .

Proof. Let  $x_1, x_2 \in [a, b]$  with  $x_1 < x_2$ . By the Mean Value Theorem, there exists  $\xi \in (x_1, x_2)$  such that

$$f(x_2) - f(x_1) = (x_2 - x_1)f'(\xi).$$

Since  $f'$  is not constant, there exists  $\xi \in (x_1, x_2)$  such that  $f'(\xi) > 0$ . Therefore,  $f(x_2) > f(x_1)$ . This shows that  $f$  is strictly increasing on  $[a, b]$ .  $\square$

(15)

$$f(x) = x^2 + 1$$

Let  $f(x) = x^2 + 1$ . Then  $f$  is a function from  $\mathbb{R}$  to  $\mathbb{R}$ . We will show that  $f$  is strictly increasing on  $\mathbb{R}$ . Let  $x_1, x_2 \in \mathbb{R}$  with  $x_1 < x_2$ . Then

$f(x_2) - f(x_1) = (x_2^2 + 1) - (x_1^2 + 1) = x_2^2 - x_1^2 = (x_2 - x_1)(x_2 + x_1)$ . Since  $x_2 - x_1 > 0$  and  $x_2 + x_1 > 0$ , it follows that  $f(x_2) > f(x_1)$ . Therefore,  $f$  is strictly increasing on  $\mathbb{R}$ .  $\square$

(16)

$$f(x) = x^3$$

Let  $f(x) = x^3$ . Then  $f$  is a function from  $\mathbb{R}$  to  $\mathbb{R}$ . We will show that  $f$  is strictly increasing on  $\mathbb{R}$ . Let  $x_1, x_2 \in \mathbb{R}$  with  $x_1 < x_2$ . Then



The two laws can be combined as follows:

$$I_t = I_o \cdot e^{-kcl} \quad (3)$$

where  $k$  is a constant.

Most colorimeters are best able to measure in terms of percentage transmission of light,  $T$ , expressed as,

$$T = \frac{I_t}{I_o} 100 \quad (4)$$

However, transmission does not vary linearly with concentration as does optical density,  $D$ , equated as,

$$D = \log_{10} \frac{I_o}{I_t} = \log_{10} \frac{100}{T} \quad (5)$$

This equation can be combined with (3) as follows:

$$D = kcl \quad \text{or,} \quad (6)$$

$$D = ecl \quad (7)$$

where  $c$  is expressed in moles per liter,  $l$  is expressed in centimeters, and  $e$  is the molar extinction coefficient. The extinction coefficient is equal to the optical density of a one-centimeter thick layer of a one molar solution of the particular substance under investigation.

Formula (5) is the one normally used with a colorimeter that reads in terms of percentage transmission of light. Under ideal conditions, and when plotted on a semi-logarithm paper against values of concentration of solution, it should

THEOREM 1. Let  $f$  be a function in  $L^p(\mathbb{R}^n)$  with  $p \geq 1$ .

$$(1) \quad \lim_{|x| \rightarrow \infty} f(x) = 0$$

if and only if  $f \in L^p(\mathbb{R}^n)$ .

It is well known that if  $f \in L^p(\mathbb{R}^n)$  then  $f(x) \rightarrow 0$  as  $|x| \rightarrow \infty$  in the sense of Lebesgue. The converse is not true.

$$(2) \quad \lim_{|x| \rightarrow \infty} \frac{1}{|x|^n} \int_{|y| \leq |x|} f(y) dy = 0$$

if and only if  $f \in L^p(\mathbb{R}^n)$  with  $p \geq 1$ . This is a stronger result than (1) and is also true for  $p = \infty$ .

$$(3) \quad \lim_{|x| \rightarrow \infty} \frac{1}{|x|^n} \int_{|y| \leq |x|} f(y) dy = 0$$

if and only if  $f \in L^p(\mathbb{R}^n)$  with  $p \geq 1$  and  $f$  is radial.

$$(4) \quad \lim_{|x| \rightarrow \infty} f(x) = 0$$

$$(5) \quad \lim_{|x| \rightarrow \infty} f(x) = 0$$

It is well known that if  $f \in L^p(\mathbb{R}^n)$  then  $f(x) \rightarrow 0$  as  $|x| \rightarrow \infty$  in the sense of Lebesgue. The converse is not true. It is also well known that if  $f \in L^p(\mathbb{R}^n)$  then  $f(x) \rightarrow 0$  as  $|x| \rightarrow \infty$  in the sense of Lebesgue. The converse is not true. It is also well known that if  $f \in L^p(\mathbb{R}^n)$  then  $f(x) \rightarrow 0$  as  $|x| \rightarrow \infty$  in the sense of Lebesgue. The converse is not true.

It is well known that if  $f \in L^p(\mathbb{R}^n)$  then  $f(x) \rightarrow 0$  as  $|x| \rightarrow \infty$  in the sense of Lebesgue. The converse is not true. It is also well known that if  $f \in L^p(\mathbb{R}^n)$  then  $f(x) \rightarrow 0$  as  $|x| \rightarrow \infty$  in the sense of Lebesgue. The converse is not true. It is also well known that if  $f \in L^p(\mathbb{R}^n)$  then  $f(x) \rightarrow 0$  as  $|x| \rightarrow \infty$  in the sense of Lebesgue. The converse is not true.

yield a perfectly straight line. The ideal conditions referred to include having a perfectly monochromatic light source and a perfect molecular solution. In the work at hand, neither condition could be realized. The colorimeter used employed a tungsten filament white light source and a series of filters which transmitted a working band of light thirty millimicrons wide (fifteen millimicrons to either side of the designated wavelength). Furthermore, as had been indicated earlier, dyes are rarely known to go into perfect molecular solution, except under special conditions.

As could be expected, in trial runs with precisely controlled concentrations of dye solutions, a curve, rather than a straight line, resulted from the use of formula (5).

As a trial, values of concentration of solution were plotted against percentage transmission light readings taken directly from the dial of the colorimeter. The resulting line was no more curved than that generated by formula (5). It was found to work very well in determining the concentration of a solution from a percentage transmission of light reading. It was then decided to construct a graph for each dye in that form. See Figures 4 through 8.

The capacity of the dyeing machine, with a one-pound package of yarn installed, was accurately determined. This volume was found to be fourteen liters. It was adhered to strictly in all dyeing runs, making due allowance for the dye solution and the salt solution to be added during the run.







A ten per cent dyeing was decided upon for all runs since this represented a maximum dyeing for sulfur dyes. This percentage on a one-pound package in fourteen liters of bath yields an initial concentration of a little more than three grams per liter. This is too dense a concentration to be read on the colorimeter. However, if the solution is diluted to one one-hundredth of its original concentration, it falls very nicely within the middle reading range of the colorimeter. This determined that the graphs would have to be constructed for a range of concentrations of from 0.050 to 0.010 gram per liter.

The calibrating solutions were prepared by weighing out, on the analytical balance, exactly one gram of dye, one gram of sodium carbonate, two grams of common salt, and from one to four grams (dependent on the dye) of crystalline sodium sulfide of reagent grade. The dye, soda ash, and sulfide were slurried with water and boiled for one minute. Three hundred milliliters of water were added and the solution was boiled for another minute. Then it was cooled to room temperature to prevent expansion when poured into the volumetric flask.

The salt was dissolved separately and then was added to the solution. The two grams of salt represent, exactly, the twenty per cent salting that each dyebath would be subjected to prior to the end of the run and the drawing of the aliquot portion sample of the dyebath for colorimetric measurement.





Sufficient water was added to the solution in the volumetric flask to make exactly one liter of solution. The solution was now an exact proportional representation of the composition of the dyebath.

Fifty milliliters of the solution were drawn with a volumetric pipette and were mixed with four hundred and fifty milliliters of water in a five hundred milliliter volumetric flask to yield a concentration of one-tenth gram per liter.

From this last as a working solution, concentrations of 0.050, 0.040, 0.030, 0.020, and 0.010 gram per liter were then prepared in a fifty milliliter volumetric flask.

This procedure of dilution was conducted to reduce the percentage of relative error to as small a value as possible.

Once the solutions were prepared, it was next necessary to determine which filter would be the most appropriate for each dye. A solution with a specific color will absorb more light of one wave length than it will of another. As an extreme example, a pure red solution will transmit one hundred per cent of a light having a pure red wavelength, regardless of its concentration. Consequently, no changes in readings could be obtained by varying the concentration of the solution. The most appropriate light is the one to which the solution will appear to be most opaque, or optically dense. The filter producing that light is the one which must be used.

the following table and notes for the purpose of the

presented table of the present and future of the  
 subject and the present and future of the  
 subject of the present.

Table of the present and future of the present

Table of the present and future of the present  
 Table of the present and future of the present  
 Table of the present and future of the present  
 Table of the present and future of the present

Table of the present and future of the present  
 Table of the present and future of the present

Table of the present and future of the present  
 Table of the present and future of the present  
 Table of the present and future of the present

Table of the present and future of the present  
 Table of the present and future of the present  
 Table of the present and future of the present

Table of the present and future of the present  
 Table of the present and future of the present  
 Table of the present and future of the present

Table of the present and future of the present  
 Table of the present and future of the present  
 Table of the present and future of the present

Table of the present and future of the present



With the 0.050 gram per liter concentration as the subject sample, readings were taken for each of eleven filters covering the entire spectrum of visible light. The filter yielding the least transmission, or the greatest absorption, of light was selected for use with the particular dye being measured. These absorption values are shown for each dye in Tables 11 through 15.

Next, with appropriate filter installed, readings of light transmission were taken for each of the concentrations of solution previously prepared. These values are shown for each of the dyes in Tables 16 through 20.

Graphs were constructed by plotting concentration of solution, in grams per liter, against corresponding percentage transmission of light readings. These graphs were used as the yardstick in determining the final concentration of the exhausted dyebath at the end of each dyeing run.

To prepare the dyebath samples, a portion of the bath was drawn from the dye chamber bleed-off with the bath circulating from the outside to the inside of the package. This sample was cooled to room temperature. Then one milliliter was pipetted from the sample and was mixed with tap water to fill a one hundred milliliter volumetric flask. This solution was then measured in the colorimeter.

In operating the colorimeter, one-centimeter cuvettes were used; tap water was used as a standard reference solution for the purpose of zeroing the instrument; and a sensitivity





of fifteen galvanometer graduations to ten transmission dial graduations was used.

Dyeing.---In every dyeing run, a ten per cent dye concentration, based on the weight of the yarn, was used. The dye was weighed out on an analytical balance to the thousandth of a gram. An equal weight of soda ash, and various weights of crystalline sodium sulfide of reagent grade were used as the alkaline solvents needed to drive the dye into aqueous solution. Twenty per cent salt, based on the weight of the yarn, was dissolved separately for later addition to the bath.

The dye, soda ash, and sulfide were slurried with water and were boiled one minute. The volume was brought up to five hundred milliliters with water, and was boiled for another minute.

The salt was made up into a five hundred milliliter solution with water and was heated until dissolved.

Prior to the actual dyeing run, each package was wet out with two grams of Duponol RA for fifteen minutes at 160° F. The wetting out bath was drained, and all residual wetting agent was rinsed off in a cold, running bath to prevent foaming during the dyeing run.

In the normal temperature runs, the bath, amounting to thirteen liters of water, was set at 190° F. The dye was added in two equal portions, one with the bath circulating from the inside of the package to the outside, and the other with the bath circulating from the outside of the package to





the inside. Dyeing was allowed to proceed for twenty minutes at 190° F. The direction of circulation of the bath was alternated every five minutes.

Salt was added to the bath over the following fifteen minute period, at the rate of about forty milliliters of solution every minute. The direction of circulation was alternated for each addition of salt.

Dyeing was allowed to proceed for an additional twenty-five minutes at 190° F. The direction of circulation was alternated every five minutes.

A sample of the exhausted dyebath was drawn at the end of the sixty minute dyeing cycle, to be measured for concentration in the colorimeter.

The bath was drained, and a running cold water rinse was applied until the bath ran clear.

Two hot rinses of 160° F. were run for ten minutes each. The package was then washed in one-half gram of Duponol RA at 120° F. for ten minutes.

The bath was drained, and a last running cold rinse was applied until the wetting agent foam and bubbles disappeared.

The package was removed from the machine and was hydro-extracted. It was dried in the hot air oven for twenty-four hours, and then it was conditioned at sixty-five per cent relative humidity and 70° F. for forty-eight hours.

the house - "I don't want to be in the house" - and the  
 as far as the situation of the house is concerned, the  
 situation is very good.

It is not clear if the house is in a good  
 or bad condition. It is not clear if the house is  
 in a good condition. It is not clear if the house is  
 in a good condition. It is not clear if the house is

in a good condition. It is not clear if the house is  
 in a good condition. It is not clear if the house is  
 in a good condition. It is not clear if the house is

in a good condition. It is not clear if the house is  
 in a good condition. It is not clear if the house is  
 in a good condition. It is not clear if the house is

in a good condition. It is not clear if the house is  
 in a good condition. It is not clear if the house is

in a good condition. It is not clear if the house is  
 in a good condition. It is not clear if the house is  
 in a good condition. It is not clear if the house is

in a good condition. It is not clear if the house is  
 in a good condition. It is not clear if the house is  
 in a good condition. It is not clear if the house is

in a good condition. It is not clear if the house is  
 in a good condition. It is not clear if the house is  
 in a good condition. It is not clear if the house is  
 in a good condition. It is not clear if the house is

The high temperature runs were conducted in precisely the same manner with the exception of the actual dyeing cycle. The bath was set at 190° F., and the dye was added in two equal portions, as before. The top of the expansion chamber was bolted into place and the temperature was raised to 250° F. within ten minutes.

Dyeing was allowed to proceed at 250° F. for fifteen minutes. The direction of circulation was alternated every five minutes.

The temperature was dropped to 190° F. by forced cooling. The top to the expansion chamber was removed, and salt was added in exactly the same manner as was done in the normal temperature run.

The run was terminated immediately after the fifteen minute salting phase, and a sample of the exhausted dyebath was taken for colorimetric measurement of concentration.

The remainder of the treatment was exactly the same as that for the normal temperature run.

The period of fifteen minutes for the high temperature phase was determined by a prolonged preliminary run, during which samples of the bath were taken as the temperature was being raised; while the temperature was being held at 250° F; as the temperature was being lowered; and after salt was added. The results of the run are shown in Table 2. During the high temperature phase, the best exhaustion (as indicated







by the highest transmission of light reading) was reached after twenty minutes at 250° F.

Because of the pressure on the system caused by the high temperature, the samples were tapped through a bleed-off on the temperature gage and pressure gage by-pass line. This included about one foot of tubing in which the dyebath remained stationary while the valve was closed. In consideration of this, it was felt that the readings from the tap must lag the actual condition of the dyebath by at least five minutes. Thus, a high temperature phase of fifteen minutes was selected.

A total of twenty dyeing runs were made, two for each dye at each temperature. It was felt that any one set of results was consistent enough to form the basis for a comparison.

Dyed Yarn Strength.--Eight break tests were made on each package of yarn for which breaking strengths had been recorded previously. The post-dyeing strengths were compared with the pre-dyeing strengths, and the change in strength for normal temperature dyeing was compared with the change in strength for high temperature dyeing. The results of these tests are shown in Table 31.

Light-Fastness Tests.--Light-fastness tests were conducted on the dyed yarn in accordance with the procedures described in the A.A.T.C.C. 1953 Yearbook (9). The samples, all together



in one group, were exposed to the arc-light of the Fade-Ometer for four twenty-hour increments. Fading was determined by visual inspection of the exposed sections under natural light. The first section to show appreciable change of shade determined the rating of the sample.

Wash-Fastness Tests.---Wash-Fastness Test Number Three, described in the A.A.T.C.C. 1953 Yearbook (10), was applied to knitted samples of the dyed yarn. In the test, bleached cotton sheeting was sewn to the back of the sample which measured two by four inches. The sample was sealed in a Mason Jar containing ten standard steel balls and one hundred milliliters of a standard solution of 0.5 per cent soap and 0.2 per cent soda ash. The sample was run in the Launder-Ometer for forty-five minutes at 160° F.

The sample was removed and washed twice in rinses of one hundred milliliters of water at 105° F. by shaking vigorously for one minute. Then it was soured in one hundred milliliters of a 0.05 per cent solution of acetic acid at 80° F. This was followed by a final rinse in one hundred milliliters of water at 80° F. The sample was hydro-extracted and ironed dry with the bleached cotton side up. Any running of the dye was determined by visual inspection of the bleached cotton.







## CHAPTER IV

## DISCUSSION OF RESULTS

Dyeing Results.—The dyes used in this experiment are listed in Table 1.

The results of the preliminary run used to determine the fifteen minute duration of the high temperature phase are shown in Table 2.

All of the normal temperature dyeing runs were sixty minutes in duration. The high temperature runs required only forty minutes to complete. This included fifteen minutes at 250° F. and fifteen minutes of salting at 190° F. The remainder of the time was used to raise and lower the temperature between these two limits. This time was a characteristic of the equipment used. It is not considered critical to the dyeing phase. It is expected that the results would be just as good if the latter time could be shortened or eliminated. This, of course, would result in an additional saving in the time required for dyeing at the higher temperature.

In every case, appreciable increase in dyebath exhaustion was experienced at the high temperature. The increase for each dye was as follows (See Tables 3 through 7):

Carbon HCF Grains	17.9%
Navy Blue 4HCF Supra	34.2%
Tan 2HCF	32.5%
Direct Blue BRCF	12.3%
Brilliant Green GCF	5.0%

# 1. Introduction

## 2. Methodology

The first part of the study is a review of the literature on the topic of

the study of

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

the study of the relationship between the two variables

Table 1. List of Dyes Used

Sulfur Dye	Light Fastness Rating	Wash Fastness Rating
Sulfogene Carbon HCF Grains	7-8	5
Sulfogene Navy Blue 4RCF Supra	4	4-5
Sulfogene Tan 2RCF	3	4-5
Sulfogene Direct Blue BRCF Conc. 200%	5-6	4-5
Sulfogene Brilliant Green GCF Extra Conc. 150%	4-5	1

Dye Manufacturer: I. E. duPont de Nemours and Company, Inc.





Table 2. Dyeing Cycle Analysis of Sulfogene Carbon RCF Grains

Time (Minutes)	Temperature (Degrees, F.)	Dyeing Phase	Transmission of Light
0	190	Enter Dye	51.0 %
5	200	Raise Temp.	51.7
8	210	"	53.1
14	220	"	52.9
21	230	"	54.9
23	240	"	53.6
33	250	High Temp.	54.0
34	250	"	54.0
35	250	"	53.8
36	250	"	53.5
37	250	"	54.1
38	250	"	54.2
43	250	"	54.9
48	250	"	55.4
53	250	"	56.3
63	250	"	56.3
67	240	Lower Temp.	55.4
74	230	"	56.7
80	220	"	57.8
85	200	Salt	59.5
108	160	Termination	62.0

## Constitution of Dyebath:

Weight of Yarn (Package Number 27)	428.2	grams
Weight of Dye	42.82	grams
Weight of Sodium Carbonate	42.82	grams
Weight of Crystalline Sodium Sulfide	85.64	grams
Weight of Sodium Chloride	8.56	grams
Volume of Dyebath	14.	liters
Concentration of Dyebath	3.059	gms/L



**Table 3. Comparison of Dyebath Exhaustions for  
Sulfogene Carbon HCF Grains**

---

<b>Average Exhaustion of High Temperature Dyebaths</b>	<b>44.7 %</b>
<b>Average Exhaustion of Normal Temperature Dyebaths</b>	<b>37.9 %</b>
<b>Gain in Exhaustion by Use of High Temperature</b>	<b>6.8 %</b>
<b>Relative Percentage Improvement by Use of High Temperature</b>	<b>17.9 %</b>
<b>Time of Normal Temperature Dyeing Cycle</b>	<b>60 mins.</b>
<b>Time of High Temperature Dyeing Cycle</b>	<b>40 mins.</b>
<b>Gain in Time by Use of High Temperature</b>	<b>20 mins.</b>
<b>Percentage Gain in Time by Use of High Temperature</b>	<b>33 %</b>

---





**Table 4. Comparison of Dyebath Exhaustions for  
Sulfogene Navy Blue 4RCF Supra**

---

<b>Average Exhaustion of High Temperature Dyebaths</b>	<b>34.9 %</b>
<b>Average Exhaustion of Normal Temperature Dyebaths</b>	<b>26.0 %</b>
<b>Gain in Exhaustion by Use of High Temperature</b>	<b>8.9 %</b>
<b>Relative Percentage Improvement by Use of High Temperature</b>	<b>34.2 %</b>
<b>Time of Normal Temperature Dyeing Cycle</b>	<b>60 mins.</b>
<b>Time of High Temperature Dyeing Cycle</b>	<b>40 mins.</b>
<b>Gain in Time by Use of High Temperature</b>	<b>20 mins.</b>
<b>Percentage Gain in Time by Use of High Temperature</b>	<b>33 %</b>

---

# THE UNIVERSITY OF MICHIGAN LIBRARIES

1000 TAPSCOTT DRIVE, ANN ARBOR, MI 48106-1000

1000

1000 TAPSCOTT DRIVE, ANN ARBOR, MI 48106-1000

1000

1000 TAPSCOTT DRIVE, ANN ARBOR, MI 48106-1000

1000

1000 TAPSCOTT DRIVE, ANN ARBOR, MI 48106-1000

1000

1000 TAPSCOTT DRIVE, ANN ARBOR, MI 48106-1000

1000

1000 TAPSCOTT DRIVE, ANN ARBOR, MI 48106-1000

1000

1000 TAPSCOTT DRIVE, ANN ARBOR, MI 48106-1000

1000

1000 TAPSCOTT DRIVE, ANN ARBOR, MI 48106-1000

1000

1000 TAPSCOTT DRIVE, ANN ARBOR, MI 48106-1000

**Table 5. Comparison of Dyebath Exhaustions for  
Sulfogene Tan 2RCF**

---

<b>Average Exhaustion of High Temperature Dyebaths</b>	<b>46.65 %</b>
<b>Average Exhaustion of Normal Temperature Dyebaths</b>	<b>35.2 %</b>
<b>Gain in Exhaustion by Use of High Temperature</b>	<b>11.45 %</b>
<b>Relative Percentage Improvement by Use of High Temperature</b>	<b>32.5 %</b>
<b>Time of Normal Temperature Cycle</b>	<b>60 mins.</b>
<b>Time of High Temperature Cycle</b>	<b>40 mins.</b>
<b>Gain in Time by Use of High Temperature</b>	<b>20 mins.</b>
<b>Percentage Gain in Time by Use of High Temperature</b>	<b>33 %</b>

---





**Table 6. Comparison of Dyebath Exhaustions for  
Sulfogene Direct Blue BRCF Cons. 200%**

---

<b>Average Exhaustion of High Temperature Dyebaths</b>	<b>35.6 %</b>
<b>Average Exhaustion of Normal Temperature Dyebaths</b>	<b>31.7 %</b>
<b>Gain in Exhaustion by Use of High Temperature</b>	<b>3.9 %</b>
<b>Relative Percentage Improvement by Use of High Temperature</b>	<b>12.3 %</b>
<b>Time of Normal Temperature Dyeing Cycle</b>	<b>60 mins.</b>
<b>Time of High Temperature Dyeing Cycle</b>	<b>40 mins.</b>
<b>Gain in Time by Use of High Temperature</b>	<b>20 mins.</b>
<b>Percentage Gain in Time by Use of High Temperature</b>	<b>33 %</b>

---



**Table 7. Comparison of Dyebath Exhaustions for Sulfogene  
Brilliant Green GCF Extra Conc. 150%**

---

<b>Average Exhaustion of High Temperature Dyebaths</b>	<b>45.0 %</b>
<b>Average Exhaustion of Normal Temperature Dyebaths</b>	<b>43.8 %</b>
<b>Gain in Exhaustion by Use of High Temperature</b>	<b>2.2 %</b>
<b>Relative Percentage Improvement by Use of High Temperature</b>	<b>5.0 %</b>
<b>Time of Normal Temperature Dyeing Cycle</b>	<b>60 mins.</b>
<b>Time of High Temperature Dyeing Cycle</b>	<b>40 mins.</b>
<b>Gain in Time by Use of High Temperature</b>	<b>20 mins.</b>
<b>Percentage Gain in Time by Use of High Temperature</b>	<b>33 %</b>

---

Table 2. Comparison of results obtained for different  
models with the same data set.

Model	Results
Model 1	Results obtained for the first model.
Model 2	Results obtained for the second model.
Model 3	Results obtained for the third model.
Model 4	Results obtained for the fourth model.
Model 5	Results obtained for the fifth model.
Model 6	Results obtained for the sixth model.
Model 7	Results obtained for the seventh model.
Model 8	Results obtained for the eighth model.
Model 9	Results obtained for the ninth model.
Model 10	Results obtained for the tenth model.
Model 11	Results obtained for the eleventh model.
Model 12	Results obtained for the twelfth model.
Model 13	Results obtained for the thirteenth model.
Model 14	Results obtained for the fourteenth model.
Model 15	Results obtained for the fifteenth model.
Model 16	Results obtained for the sixteenth model.
Model 17	Results obtained for the seventeenth model.
Model 18	Results obtained for the eighteenth model.
Model 19	Results obtained for the nineteenth model.
Model 20	Results obtained for the twentieth model.
Model 21	Results obtained for the twenty-first model.
Model 22	Results obtained for the twenty-second model.
Model 23	Results obtained for the twenty-third model.
Model 24	Results obtained for the twenty-fourth model.
Model 25	Results obtained for the twenty-fifth model.
Model 26	Results obtained for the twenty-sixth model.
Model 27	Results obtained for the twenty-seventh model.
Model 28	Results obtained for the twenty-eighth model.
Model 29	Results obtained for the twenty-ninth model.
Model 30	Results obtained for the thirtieth model.



The shades of the high temperature products were at least equal to those of the normal temperature products. In some cases they were slightly heavier, or darker, to a barely noticeable measure. In the case of the Brilliant Green, the high temperature products had a distinctly bluer chroma. The Tan dye produced a greener, or olive, cast at high temperature, in contrast to the redder or yellower chroma of the normal temperature product. The other dyes produced the same color at both temperatures.

An effort was made to compare the penetrations of the dyes by means of microscopic cross-sections. The thinnest cross-sectional slice that could be made with the equipment on hand was two and one-half microns. The dye was too dense, even in that thin a cross-section, to transmit sufficient working light for microscopic inspection. The only general conclusion that can be drawn is that at least good penetration was obtained.

Light-Fastness Results.--All of the dyes broke within sixty hours. For each dye, the high temperature and normal temperature products broke at exactly the same time. The time required to produce fading in each dye was as follows:

Carbon BCF Grains	60 hours
Navy Blue 4RCF Supra	60 hours
Tan 2RCF	20 hours
Direct Blue BRCF	60 hours
Brilliant Green GCF	60 hours



Wash-Fastness Results.--All of the dyes bled into the bleached cotton backing during the Number Three Wash Test. Neither the high temperature nor the normal temperature product bled appreciably more than the other. However, the high temperature product did appear to retain more of its original depth of shade than did the normal temperature product. This would be due to the heavier take-on of dye that we were able to achieve at the higher temperature.

In evaluating the absolute results of the light and wash fastness tests, we must remember that none of the dyeings was after-treated. It is known that sulfur dyes derive some resistance to light and washing from treatment with copper sulfate or acetic acid. After-treatment was not resorted to so that any differences in results could be ascribed directly and purely to the difference in temperatures.

Residual Yarn Strength Results.--Neither the high temperature product nor the normal temperature product appeared to lose any significant measure of strength after being dyed. See Table 31 for detailed results. As had been pointed out earlier, the yarn was very irregular in strength. Though Table 31 shows some increase in strength, it is not safe to assume that such was the actual case. This must be ascribed to the inherent variation in the yarn.







## CHAPTER V

### CONCLUSIONS

All of the remarks that follow, until otherwise qualified, must be applied strictly to the five dyes employed in this investigation.

High temperatures can be used successfully and profitably to apply the sulfur dye on cotton yarn. A one-third saving in time and an increase in exhaustion of from 5.0 to 34.2 per cent can be had concurrently by using this method. This saving in time refers solely to the actual dyeing cycle only. The resulting dyed products are, in every way, as good as those of the normal temperature process. Neither the dye nor the yarn suffers significantly from exposure to the higher temperature.

Since a one-third saving in time and an increase in exhaustion were experienced concurrently, it must be assumed that an exhaustion equal to that of the normal temperature process can be achieved with an even greater saving in time. The corresponding high temperature dyeing time, of course, would have to be determined empirically for each dye.

Though the qualitative results of this work may, in some measure, be an indication of the behavior to be expected of the remainder of the dyes in this class, the quantitative

# THE HISTORY

## OF THE

OF THE HISTORY OF THE UNITED STATES OF AMERICA, FROM THE FIRST SETTLEMENTS TO THE PRESENT TIME. BY JAMES M. SMITH, ESQ. VOL. I.

THE HISTORY OF THE UNITED STATES OF AMERICA, FROM THE FIRST SETTLEMENTS TO THE PRESENT TIME. BY JAMES M. SMITH, ESQ. VOL. I. PART I. THE DISCOVERY AND SETTLEMENT OF THE COUNTRY. THE FIRST PART OF THE HISTORY OF THE UNITED STATES OF AMERICA, FROM THE FIRST SETTLEMENTS TO THE PRESENT TIME. BY JAMES M. SMITH, ESQ. VOL. I. PART I. THE DISCOVERY AND SETTLEMENT OF THE COUNTRY. THE FIRST PART OF THE HISTORY OF THE UNITED STATES OF AMERICA, FROM THE FIRST SETTLEMENTS TO THE PRESENT TIME. BY JAMES M. SMITH, ESQ. VOL. I. PART I. THE DISCOVERY AND SETTLEMENT OF THE COUNTRY.

THE HISTORY OF THE UNITED STATES OF AMERICA, FROM THE FIRST SETTLEMENTS TO THE PRESENT TIME. BY JAMES M. SMITH, ESQ. VOL. I. PART I. THE DISCOVERY AND SETTLEMENT OF THE COUNTRY. THE FIRST PART OF THE HISTORY OF THE UNITED STATES OF AMERICA, FROM THE FIRST SETTLEMENTS TO THE PRESENT TIME. BY JAMES M. SMITH, ESQ. VOL. I. PART I. THE DISCOVERY AND SETTLEMENT OF THE COUNTRY. THE FIRST PART OF THE HISTORY OF THE UNITED STATES OF AMERICA, FROM THE FIRST SETTLEMENTS TO THE PRESENT TIME. BY JAMES M. SMITH, ESQ. VOL. I. PART I. THE DISCOVERY AND SETTLEMENT OF THE COUNTRY.

results should not be taken as either criteria or limits of performance for the class as a whole. There is no reason to assume that either the best or the worst performing member of this class was used in this experiment. The range of performances of the dyes used is so wide as not to preclude the possibility that one or more members of the class will not perform successfully at the higher temperature. By the same token, it is not impossible that there is one or more members which will be superior to any of the dyes used.





## CHAPTER VI

### RECOMMENDATIONS

Investigation into the behavior of a small group of dyes under special conditions is valuable only in determining the possible modes of behavior that may be expected of the whole class to which the dyes belong.

It has been shown only that some sulfur dyes can be used successfully at a high temperature. Whether all sulfur dyes will behave similarly remains to be determined.

Before the dyeing industry can proceed to use sulfur dyes at a high temperature, it should be equipped with empirical information on which of the dyes can be so used and on the best procedures for applying them. The only authoritative sources for this data are the dyestuff manufacturers.

Therefore, it is recommended that the dyestuff manufacturers collect such information and make it available to the dyeing industry so that it can take advantage of the economies and other benefits that may be realized from the use of high temperatures.



**APPENDIX**





Table 8. Yarn Number of Scoured, Undyed Cotton Yarn

Sample Number	Weight in Grains of 120 Yards	Yarn Number
1	57.8	17.31
2	61.5	16.25
3	65.5	15.25
4	61.5	16.25
5	63.8	15.65
6	63.9	15.64
7	59.2	16.89
8	68.3	14.63
9	58.1	17.21
10	61.6	16.23
11	65.6	15.22
12	61.7	16.21
13	64.0	15.61
14	64.0	15.61
15	59.8	16.72
16	68.6	14.59
17	57.9	17.29
18	61.4	16.27
19	65.4	15.38
20	61.3	16.29
21	63.7	15.69
22	63.8	15.66
23	59.1	16.91
24	62.2	14.67
25	59.7	16.77
Total		400.10
Average		16.0



**Table 9. Twist of Scoured, Undyed Cotton Yarn  
in Turns per Inch**

**Direction of Twist: "Z"**

<b>Sample Number</b>	<b>Turns per Inch</b>
1	18.10
2	17.50
3	19.75
4	16.80
5	18.10
6	16.50
7	17.25
8	17.60
9	19.60
10	18.85
11	18.20
12	19.85
13	18.55
14	16.50
15	18.20
<b>Total</b>	<b>271.95</b>
<b>Average</b>	<b>18.10</b>

THE UNIVERSITY OF CHICAGO  
 LIBRARY OF THE DIVISION OF THE PHYSICAL SCIENCES  
 5708 S. UNIVERSITY AVENUE, CHICAGO, ILL. 60637

NOV 19 1967

Date Recd.	Amount
10/10/67 100.00	100.00
11/10/67 100.00	100.00



**Table 10. Single Strand Breaking Strength of Scoured,  
Undyed Cotton Yarn**

<b>Sample Number</b>	<b>Strength (Pounds)</b>	<b>Sample Number</b>	<b>Strength (Pounds)</b>
1	1.30	21	1.42
2	1.25	22	1.43
3	1.25	23	1.52
4	1.10	24	1.62
5	1.13	25	1.54
6	1.20	26	1.10
7	1.08	27	1.00
8	1.02	28	0.90
9	1.20	29	0.96
10	1.04	30	0.92
11	1.14	31	1.14
12	1.12	32	1.10
13	1.00	33	1.14
14	1.00	34	1.20
15	0.96	35	1.16
16	1.20	36	1.36
17	1.00	37	1.14
18	0.92	38	1.10
19	1.22	39	1.00
20	1.12	40	0.92
<b>Total</b>			<b>45.99</b>
<b>Average</b>			<b>1.15</b>



**Table 11. Light Absorption Percentages for  
Sulfogene Carbon HCF Grains**

<b>Value of Monochromatic Filter (millimicrons)</b>	<b>Color</b>	<b>Percentage Transmission of Light</b>	<b>Percentage Absorption of Light</b>
390	Violet	7.0 %	93.0 %
440	Blue	12.3	87.7
465	Blue	14.8	85.2
490	Green	15.6	84.4
515	Green	15.5	84.5
550	Green	14.7	85.3
575	Amber	13.1	86.9
595	Orange	14.0	86.0
620	Red	13.5	86.5
640	Red	12.4	87.6
660	Red	12.0	88.0

**Composition of Solution:**

1 gram of dye  
 1 gram of sodium carbonate  
 2 grams of crystalline sodium sulfide  
 2 grams of sodium chloride  
 Sufficient tap water to make one liter  
 of solution

The above solution was then diluted to yield a concentration of one-tenth of a gram of dye per liter of solution.





**Table 12. Light Absorption Percentages for  
Sulfogene Navy Blue 4RCP Supra**

<b>Value of Monochromatic Filter (millimicrons)</b>	<b>Color</b>	<b>Percentage Transmission of Light</b>	<b>Percentage Absorption of Light</b>
390	Violet	55.8 %	44.2 %
440	Blue	59.4	40.6
465	Blue	54.6	45.4
490	Green	39.7	60.3
515	Green	31.4	68.6
550	Green	21.5	78.5
575	Amber	28.0	72.0
595	Orange	32.6	67.4
620	Red	36.4	63.6
640	Red	44.1	55.9
660	Red	49.7	50.3

**Composition of Solution:**

1 gram of dye  
 1 gram of sodium carbonate  
 4 grams of crystalline sodium sulfide  
 2 grams of sodium chloride  
 Sufficient tap water to make one liter  
 of solution

The above solution was then diluted to yield a concentration of five hundredths of a gram of dye per liter of solution.

THE UNIVERSITY OF CHICAGO PRESS  
530 N. Dearborn Ave. Chicago, Ill. 60610-5708

Author's name Last, first	Author's name Last, first	Title	Year published (month and day)
A. B. C.	A. B. C.	1997	1997
A. B. C.	A. B. C.	1998	1998
A. B. C.	A. B. C.	1999	1999
A. B. C.	A. B. C.	2000	2000
A. B. C.	A. B. C.	2001	2001
A. B. C.	A. B. C.	2002	2002
A. B. C.	A. B. C.	2003	2003
A. B. C.	A. B. C.	2004	2004
A. B. C.	A. B. C.	2005	2005
A. B. C.	A. B. C.	2006	2006
A. B. C.	A. B. C.	2007	2007
A. B. C.	A. B. C.	2008	2008
A. B. C.	A. B. C.	2009	2009
A. B. C.	A. B. C.	2010	2010
A. B. C.	A. B. C.	2011	2011
A. B. C.	A. B. C.	2012	2012
A. B. C.	A. B. C.	2013	2013
A. B. C.	A. B. C.	2014	2014
A. B. C.	A. B. C.	2015	2015
A. B. C.	A. B. C.	2016	2016
A. B. C.	A. B. C.	2017	2017
A. B. C.	A. B. C.	2018	2018
A. B. C.	A. B. C.	2019	2019
A. B. C.	A. B. C.	2020	2020
A. B. C.	A. B. C.	2021	2021
A. B. C.	A. B. C.	2022	2022
A. B. C.	A. B. C.	2023	2023
A. B. C.	A. B. C.	2024	2024
A. B. C.	A. B. C.	2025	2025
A. B. C.	A. B. C.	2026	2026
A. B. C.	A. B. C.	2027	2027
A. B. C.	A. B. C.	2028	2028
A. B. C.	A. B. C.	2029	2029
A. B. C.	A. B. C.	2030	2030

Copyright © 2023 by The University of Chicago Press

ALL RIGHTS RESERVED  
No part of this publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, recording, or by any information storage and retrieval system, without permission in writing from the University of Chicago Press.

This publication is a book in the series of books on the history of the world, and is published by the University of Chicago Press.

**Table 13. Light absorption Percentages for  
Sulfogene Tan 2RCF**

<b>Value of Monochromatic Filters (millimicrons)</b>	<b>Color</b>	<b>Percentage Transmission of Light</b>	<b>Percentage Absorption of Light</b>
390	Violet	36.9 %	63.1 %
440	Blue	39.8	60.2
465	Blue	48.3	51.7
490	Green	57.1	42.9
515	Green	61.8	38.2
550	Green	80.4	19.6
575	Amber	91.3	8.7
595	Orange	94.6	5.4
620	Red	96.0	4.0
640	Red	96.8	3.2
660	Red	97.2	2.8

**Composition of Solution:**

1 gram of dye  
 1 gram of sodium carbonate  
 4 grams of crystalline sodium sulfide  
 2 grams of sodium chloride  
 Sufficient tap water to make one liter  
 of solution

The above solution was then diluted to yield a concentration of five hundredths of a gram of dye per liter of solution.

# TABLE 1. SUMMARY OF RESULTS FOR THE STUDY OF THE EFFECTS OF THE 1974-75 DROUGHT ON THE GROWTH OF THE COMMON PEA (*Pisum sativum* L.)

PLANTING DATE	PLANTING DENSITY	PLANTING METHOD	PLANTING DATE
1.1.75	1.1.75	1.1.75	1.1.75
1.2.75	1.2.75	1.2.75	1.2.75
1.3.75	1.3.75	1.3.75	1.3.75
1.4.75	1.4.75	1.4.75	1.4.75
1.5.75	1.5.75	1.5.75	1.5.75
1.6.75	1.6.75	1.6.75	1.6.75
1.7.75	1.7.75	1.7.75	1.7.75
1.8.75	1.8.75	1.8.75	1.8.75
1.9.75	1.9.75	1.9.75	1.9.75
1.10.75	1.10.75	1.10.75	1.10.75
1.11.75	1.11.75	1.11.75	1.11.75

PLANTING DATE: 1.1.75, 1.2.75, 1.3.75, 1.4.75, 1.5.75, 1.6.75, 1.7.75, 1.8.75, 1.9.75, 1.10.75, 1.11.75

PLANTING DENSITY: 1.1.75, 1.2.75, 1.3.75, 1.4.75, 1.5.75, 1.6.75, 1.7.75, 1.8.75, 1.9.75, 1.10.75, 1.11.75

PLANTING METHOD: 1.1.75, 1.2.75, 1.3.75, 1.4.75, 1.5.75, 1.6.75, 1.7.75, 1.8.75, 1.9.75, 1.10.75, 1.11.75



**Table 14. Light Absorption Percentages for  
Sulfogene Direct Blue BRCF Conc. 200%**

<b>Value of Monochromatic Filter (millimicrons)</b>	<b>Color</b>	<b>Percentage Transmission of Light</b>	<b>Percentage Absorption of Light</b>
390	Violet	48.9 %	51.1 %
440	Blue	54.7	45.3
465	Blue	53.4	46.6
490	Green	45.1	54.9
515	Green	39.7	60.3
550	Green	33.1	66.9
575	Amber	36.9	63.1
595	Orange	40.0	60.0
620	Red	42.7	57.3
640	Red	47.4	52.6
660	Red	51.3	48.7

**Composition of Solution:**

1 gram of dye  
 1 gram of sodium carbonate  
 3 grams of crystalline sodium sulfide  
 2 grams of sodium chloride  
 Sufficient tap water to make one liter  
 of solution

The above solution was then diluted to yield a concentration of five hundredths of a gram of dye per liter of solution.



**Table 15. Light Absorption Percentages for Sulfogene  
Brilliant Green GCF Extra Conc. 150%**

<b>Value of Monochromatic Filter (millimicrons)</b>	<b>Color</b>	<b>Percentage Transmission of Light</b>	<b>Percentage Absorption of Light</b>
390	Violet	61.6 %	38.4 %
440	Blue	73.8	26.2
465	Blue	79.7	20.3
490	Green	81.4	18.6
515	Green	80.0	20.0
550	Green	67.7	32.3
575	Amber	55.4	44.6
595	Orange	52.0	48.0
620	Red	52.4	47.6
640	Red	55.5	44.5
660	Red	58.5	41.5

**Composition of Solution:**

1 gram of dye  
 1 gram of sodium carbonate  
 1 gram of crystalline sodium sulfide  
 2 grams of sodium chloride  
 Sufficient tap water to make one liter  
 of solution

The above solution was then diluted to yield a concentration of five hundredths of a gram of dye per liter of solution.

TABLE 1. Summary of the results of the analysis of variance for the effect of the treatment on the response of the subjects to the treatment.

Source of variation	Sum of squares	D.F.	Mean square	F-value
Treatment	10.00	1	10.00	1.00
Block	10.00	1	10.00	1.00
Subject	10.00	1	10.00	1.00
Treatment x Block	10.00	1	10.00	1.00
Treatment x Subject	10.00	1	10.00	1.00
Block x Subject	10.00	1	10.00	1.00
Treatment x Block x Subject	10.00	1	10.00	1.00
Error	10.00	1	10.00	1.00
Total	10.00	1	10.00	1.00

TABLE 2. Summary of the results of the analysis of variance for the effect of the treatment on the response of the subjects to the treatment.

Source of variation	Sum of squares	D.F.	Mean square	F-value
Treatment	10.00	1	10.00	1.00
Block	10.00	1	10.00	1.00
Subject	10.00	1	10.00	1.00
Treatment x Block	10.00	1	10.00	1.00
Treatment x Subject	10.00	1	10.00	1.00
Block x Subject	10.00	1	10.00	1.00
Treatment x Block x Subject	10.00	1	10.00	1.00
Error	10.00	1	10.00	1.00
Total	10.00	1	10.00	1.00

TABLE 3. Summary of the results of the analysis of variance for the effect of the treatment on the response of the subjects to the treatment.



**Table 16. Light Transmission Percentages for  
Sulfogene Carbon HCF Grains**

Concentration in Grams per Liter	Percentage Transmission
0.01	76.6
0.02	59.6
0.03	46.3
0.04	36.4
0.05	27.6

**Composition of Solution:**

1 gram of dye  
1 gram of sodium carbonate  
2 grams of crystalline sodium sulfide  
2 grams of sodium chloride  
Sufficient tap water to make one liter  
of solution

The solution was then diluted to yield the working concentrations listed above.

**Monochromatic Filter Used:** Violet - 390 millimicrons

**Scale Ratio Used:** 15 Galvanometer Graduations to  
10 Transmission Dial Graduations

Table 10. (continued) (continued)

Parameter	Value
1.00	1.00
1.00	1.00
1.00	1.00
1.00	1.00
1.00	1.00

Continued on next page

1.00 1.00 1.00  
1.00 1.00 1.00  
1.00 1.00 1.00  
1.00 1.00 1.00  
1.00 1.00 1.00  
1.00 1.00 1.00

The data are presented in Table 10. The data are presented in Table 10.

Continued on next page

1.00 1.00 1.00  
1.00 1.00 1.00  
1.00 1.00 1.00  
1.00 1.00 1.00

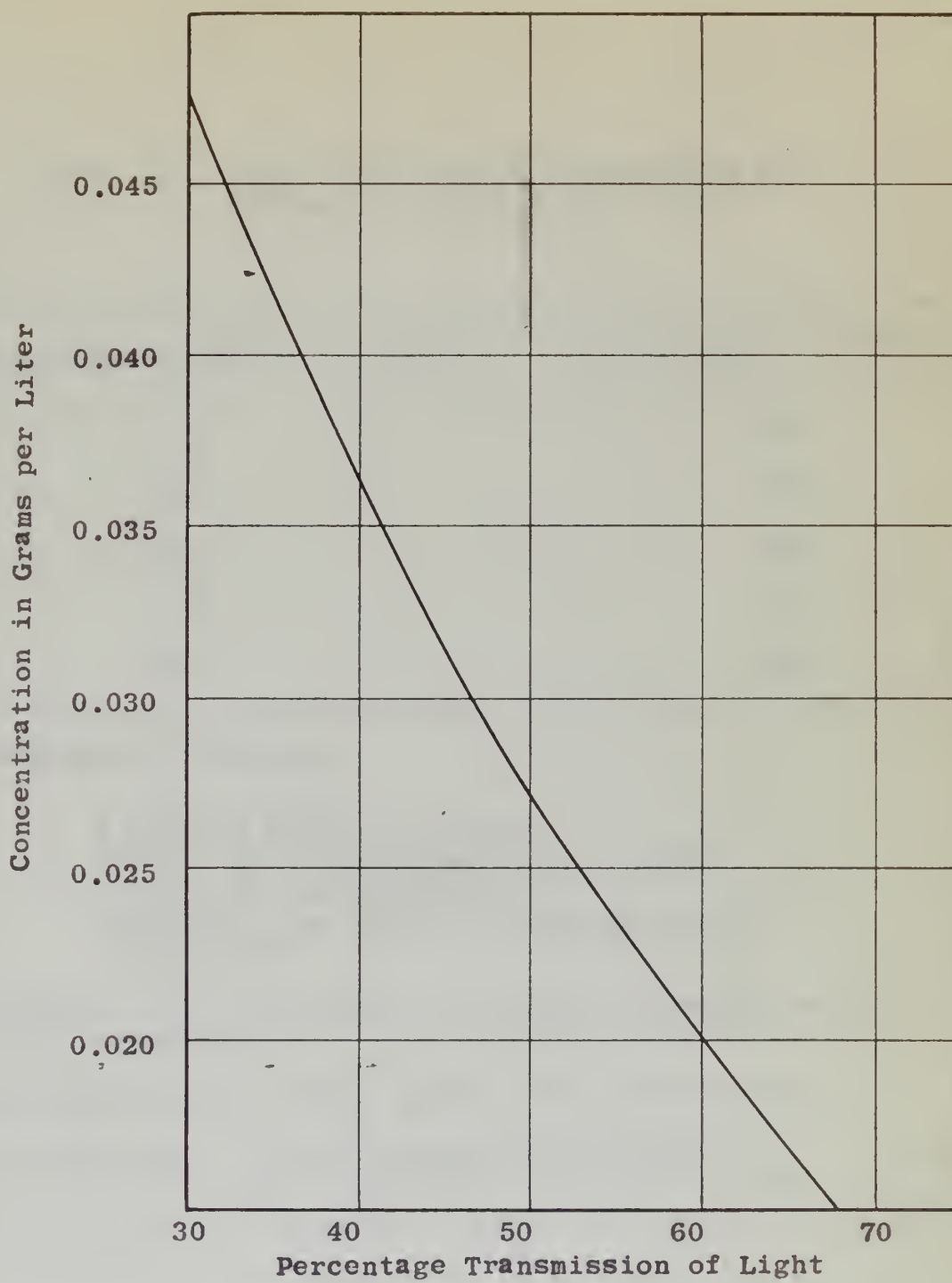


Fig. 4 Sulfogene Carbon HCF Grains  
Concentration vs. Percentage  
Light Transmission.





Table 17. Light Transmission Percentages for  
Sulfogene Navy Blue 4RCF Supra

Concentration in Grams per Liter	Percentage Transmission
0.01	73.1
0.02	54.0
0.03	39.5
0.04	28.9
0.05	21.5

Composition of Solution:

1 gram of dye  
1 gram of sodium carbonate  
4 grams of crystalline sodium sulfide  
2 grams of sodium chloride  
Sufficient tap water to make one liter  
of solution

The solution was then diluted to yield the working concentrations listed above.

Monochromatic Filter Used: Green - 550 millimicrons

Scale Ratio Used: 15 Galvanometer Graduations to  
10 Transmission Dial Graduations



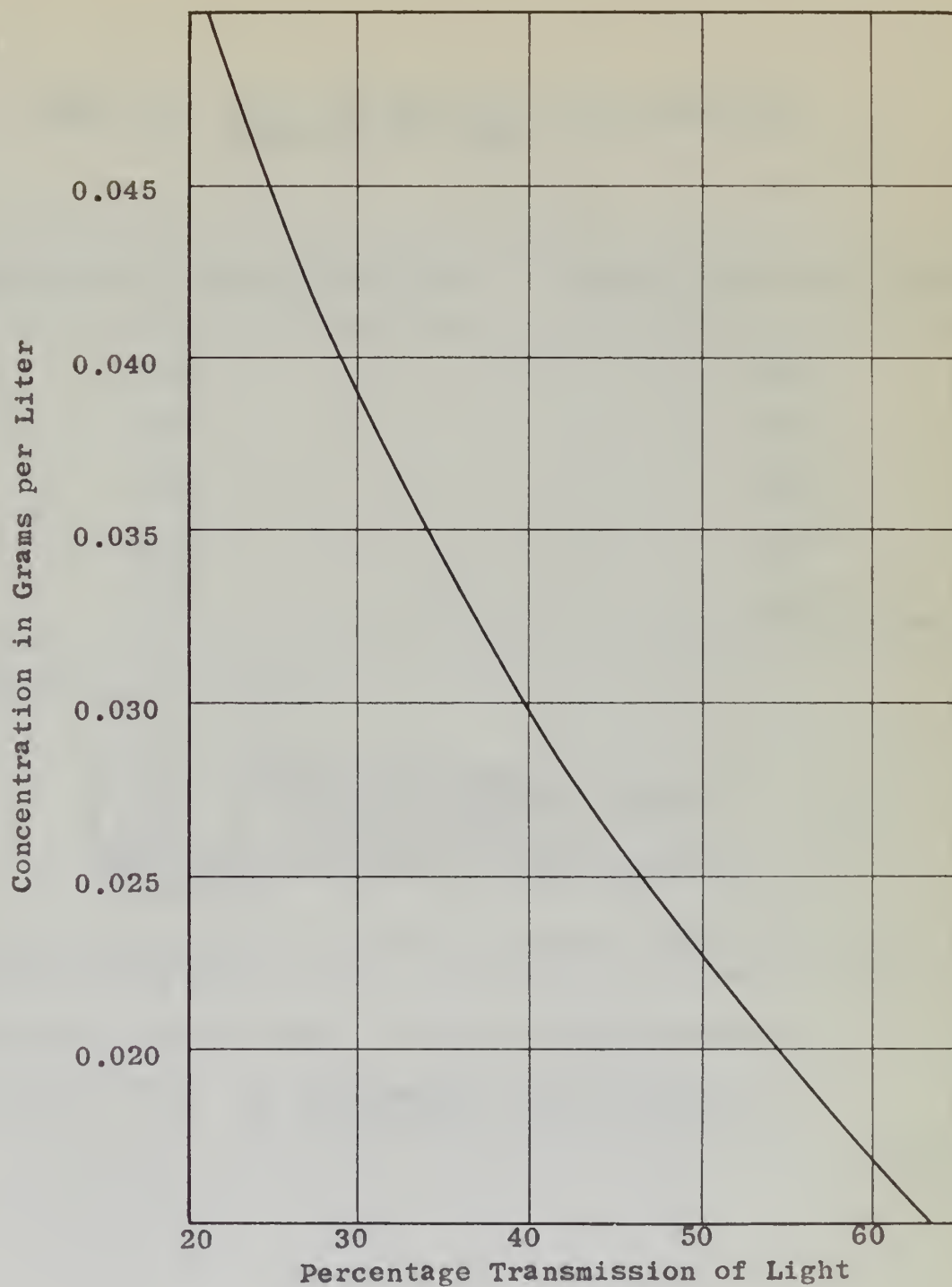


Fig. 5 Sulfogene Navy Blue 4RCF Supra  
Concentration vs. Percentage  
Transmission of Light





**Table 18. Light Transmission Percentages for  
Sulfogens Tan 2MCF**

Concentration in Grams per Liter	Percentage Transmission
0.01	83.1
0.02	68.8
0.03	56.1
0.04	45.5
0.05	36.9

**Composition of Solution:**

1 gram of dye  
1 gram of sodium carbonate  
4 grams of crystalline sodium sulfide  
2 grams of sodium chloride  
Sufficient tap water to make one liter  
of solution

The solution was then diluted to yield the working concentrations listed above.

**Monochromatic Filter Used:** Violet - 390 millimicrons

**Scale Ratio Used:** 15 Galvanometer Graduations to  
10 Transmission Dial Graduations



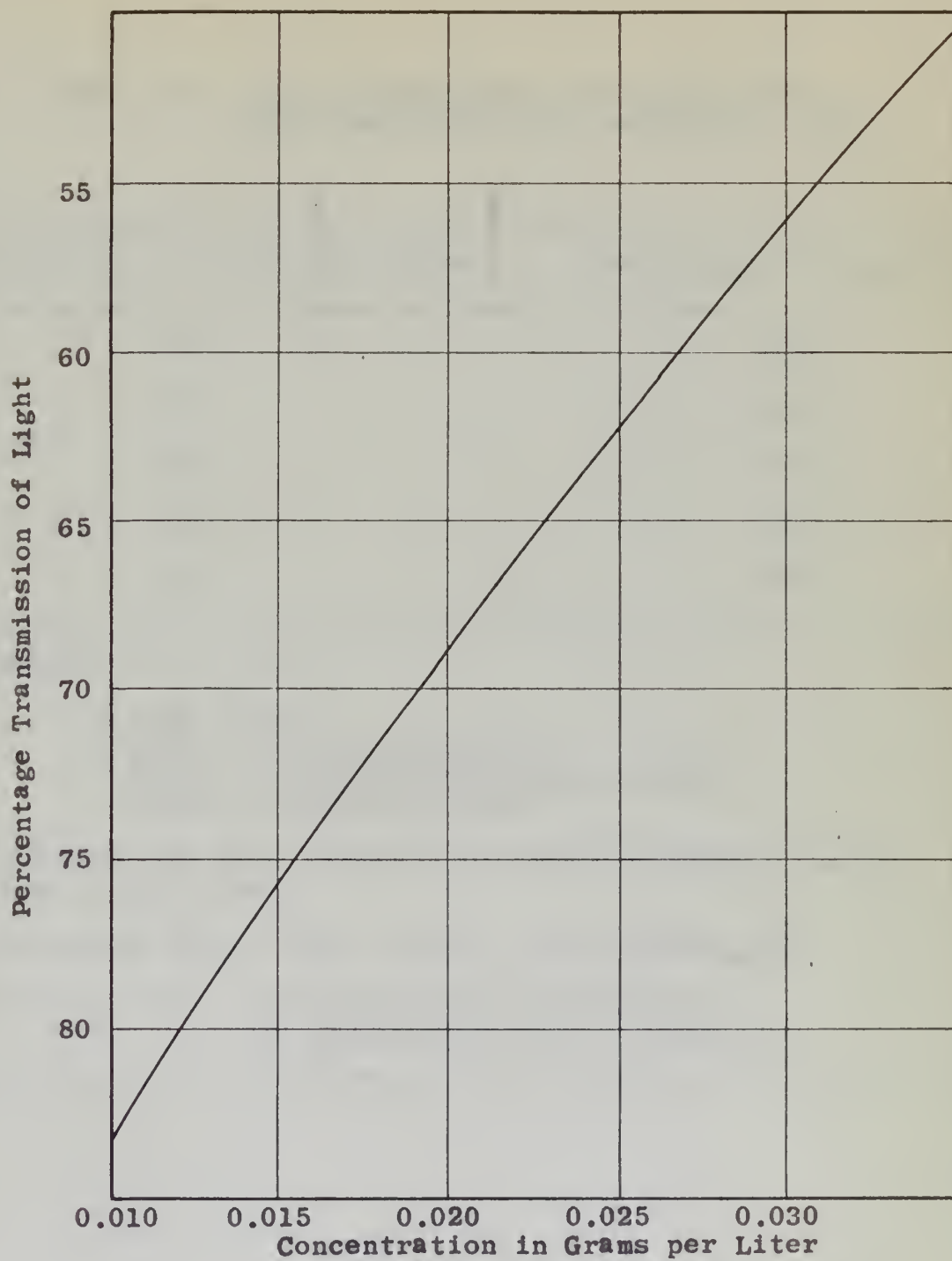


Fig. 6 Sulfolene Tan 2RCF  
Concentration vs. Percentage  
Transmission of Light





Table 19. Light Transmission Percentages for  
Sulfogene Direct Blue BRCF Conc. 200%

Concentration in Grams per Liter	Percentage Transmission
0.01	79.2
0.02	64.0
0.03	51.2
0.04	41.0
0.05	33.1

Composition of Solution:

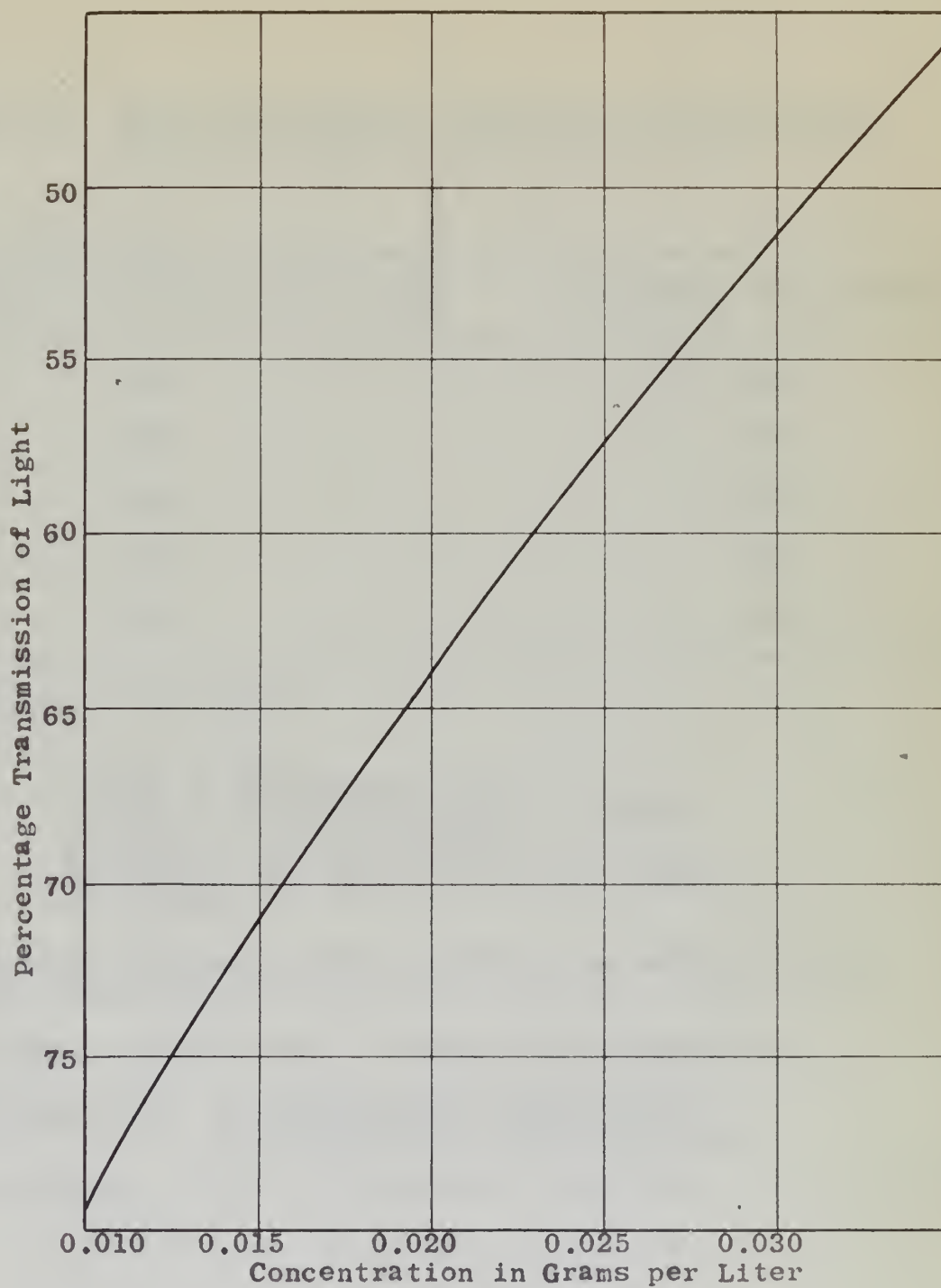
1 gram of dye  
1 gram of sodium carbonate  
3 grams of crystalline sodium sulfide  
2 grams of sodium chloride

The solution was then diluted to yield the working concentrations listed above.

Monochromatic Filter Used: Green - 550 millimicrons

Scale Ratio Used: 15 Galvanometer Graduations to  
10 Transmission Dial Graduations





**Fig. 7** Sulfolene Direct Blue BRCF Conc. 200%  
Concentration vs. Percentage  
Transmission of Light





**Table 20. Light Transmission Percentages for Sulfogens  
Brilliant Green GCF Extra Conc. 150%**

Concentration in Grams per Liter	Percentage Transmission
0.01	88.0
0.02	77.1
0.03	67.5
0.04	58.9
0.05	51.5

**Composition of Solution:**

1 gram of dye  
1 gram of sodium carbonate  
1 gram of crystalline sodium sulfide  
2 grams of sodium chloride  
Sufficient tap water to make one liter  
of solution

The solution was then diluted to yield the working concentrations listed above.

**Monochromatic Filter Used:** Orange - 595 millimicrons

**Scale Ratio Used:** 15 Galvanometer Graduations to  
10 Transmission Dial Graduations

THESE ARE THE ONLY DOCUMENTS RELATING TO THE  
 CASE, AND ARE NOT TO BE REPRODUCED

REMARKS	DATE
1.00	19.0
1.77	19.0
1.79	19.0
1.80	19.0
1.20	19.0

REMARKS TO BE MADE

1. The value of  
 the property is not  
 sufficient to cover the  
 cost of the property  
 and the value of the  
 property is not sufficient  
 to cover the cost of the  
 property.

THESE ARE THE ONLY DOCUMENTS RELATING TO THE  
 CASE, AND ARE NOT TO BE REPRODUCED

REMARKS TO BE MADE

1. The value of  
 the property is not  
 sufficient to cover the  
 cost of the property  
 and the value of the  
 property is not sufficient  
 to cover the cost of the  
 property.

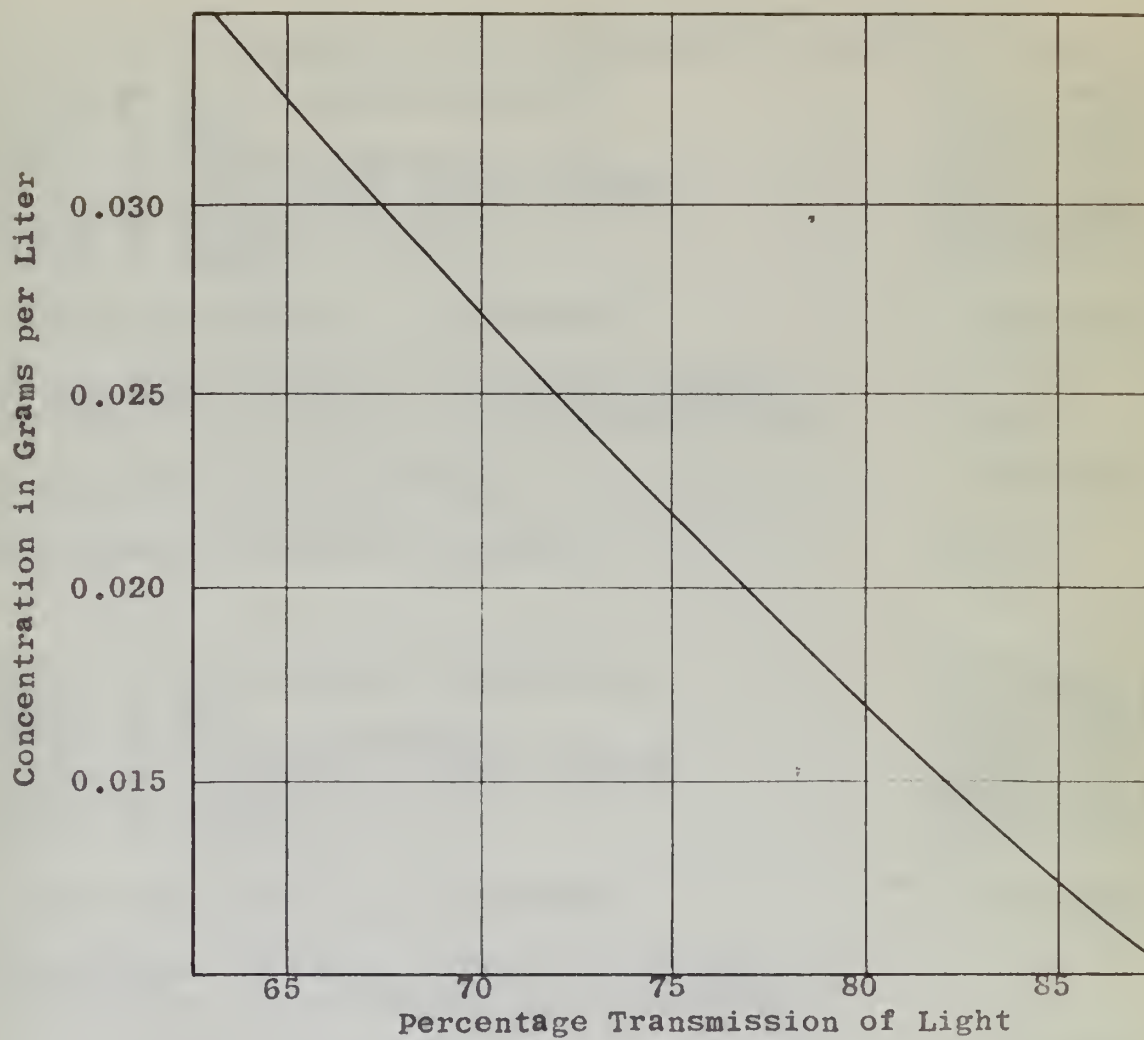


Fig. 8 Sulfogene Brilliant Green GCF Extra ,  
Conc. 150% Concentration vs. Per-  
centage Transmission of Light





**Table 21. Dyebath Exhaustion for Sulfogene Carbon HCF Grains at 190 Degrees, Fahrenheit, for Sixty Minutes**

---

Weight of Yarn (package Number 1)	426.00 grams
Weight of Dye	42.60 grams
Weight of Sodium Carbonate	42.60 grams
Weight of Crystalline Sodium Sulfide	85.20 grams
Weight of Sodium Chloride	85.20 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	3.04 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	60.9 %
Corresponding Concentration	1.92 gms/L
Percentage Exhaustion of Dyebath	36.8 %
Weight of Yarn (package Number 14)	438.00 grams
Weight of Dye	43.80 grams
Weight of Sodium Carbonate	43.80 grams
Weight of Crystalline Sodium Sulfide	87.60 grams
Weight of Sodium Chloride	87.60 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	3.13 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	61.0 %
Corresponding Concentration	1.91 gms/L
Percentage Exhaustion of Dyebath	39.0 %
Average Exhaustion of Dyebaths	37.9 %

---



**Table 22. Dyebath Exhaustion for Sulfogene Carbon NCF Grains at High Temperature**

**Time of Dyeing: Forty minutes (including 15 minutes at 250 degrees, F.)**

---

Weight of Yarn (Package Number 2)	414.750 grams
Weight of Dye	41.475 grams
Weight of Sodium Carbonate	41.475 grams
Weight of Crystalline Sodium Sulfide	82.950 grams
Weight of Sodium Chloride	82.950 grams
Volume of Dyebath	14.000 liters
Initial Concentration of Dyebath	2.96 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	65.2 %
Corresponding Concentration	1.65 gms/L
Percentage Exhaustion of Dyebath	44.3 %
Weight of Yarn (Package Number 12)	436.70 grams
Weight of Dye	43.67 grams
Weight of Sodium Carbonate	43.67 grams
Weight of Crystalline Sodium Sulfide	87.34 grams
Weight of Sodium Chloride	87.34 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	3.12 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	64.3 %
Corresponding Concentration	1.71 gms/L
Percentage Exhaustion of Dyebath	45.2 %
Average Exhaustion of Dyebaths	44.7 %

---



THE UNITED STATES OF AMERICA, DISTRICT COURT  
SOUTHERN DISTRICT OF NEW YORK

IN SENATE OF JULY TWENTY-NINE, 1957  
1:57 PM, NEW YORK

Salary, \$10,000	(1) Salary payable by the United States
Health, \$10,000	to the United States
Travel, \$10,000	to the United States
Living, \$10,000	to the United States
Supplies, \$10,000	to the United States
Other, \$10,000	to the United States

Approved by the President of the United States

Witness my hand and the Great Seal of the United States  
this 29th day of July, 1957.

Done at the City of New York, New York, this 29th day of July, 1957.

Witness my hand and the Great Seal of the United States

Salary, \$10,000	(2) Salary payable by the United States
Health, \$10,000	to the United States
Travel, \$10,000	to the United States
Living, \$10,000	to the United States
Supplies, \$10,000	to the United States
Other, \$10,000	to the United States

Approved by the President of the United States

Witness my hand and the Great Seal of the United States  
this 29th day of July, 1957.

Done at the City of New York, New York, this 29th day of July, 1957.

Witness my hand and the Great Seal of the United States

Approved by the President of the United States



**Table 23. Dyebath Exhaustion for Sulfogene Navy Blue 4RCF  
Supra at 190 Degrees, Fahrenheit, for Sixty  
Minutes**

---

Weight of Yarn (Package Number 3)	426.40 grams
Weight of Dye	42.64 grams
Weight of Sodium Carbonate	42.64 grams
Weight of Crystalline Sodium Sulfide	170.56 grams
Weight of Sodium Chloride	85.28 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	3.046 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	49.7 %
Corresponding Concentration	2.275 gms/L
Percentage Exhaustion of Dyebath	25.3 %
Weight of Yarn (Package Number 15)	456.00 grams
Weight of Dye	45.60 grams
Weight of Sodium Carbonate	45.60 grams
Weight of Crystalline Sodium Sulfide	182.40 grams
Weight of Sodium Chloride	91.20 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	3.257 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	47.9 %
Corresponding Concentration	2.388 gms/L
Percentage Exhaustion of Dyebath	26.7 %
Average Exhaustion of Dyebaths	26.0 %

---



**Table 24. Dyebath Exhaustion for Sulfogene Navy Blue 4ECF  
Supra at High Temperature**

**Time of Dyeing: Forty minutes (including 15 minutes at  
250 degrees, F.)**

---

Weight of Yarn (Package Number 4)	420.1 grams
Weight of Dye	42.01 grams
Weight of Sodium Carbonate	42.01 grams
Weight of Crystalline Sodium Sulfide	168.04 grams
Weight of Sodium Chloride	84.02 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	3.001 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	54.8 %
Corresponding Concentration	1.945 gms/L
Percentage Exhaustion of Dyebath	35.2 %
Weight of Yarn (Package Number 16)	440.1 grams
Weight of Dye	44.01 grams
Weight of Sodium Carbonate	44.01 grams
Weight of Crystalline Sodium Sulfide	176.04 grams
Weight of Sodium Chloride	88.02 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	3.144 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	53.0 %
Corresponding Concentration	2.058 gms/L
Percentage Exhaustion of Dyebath	34.5 %
Average Exhaustion of Dyebaths	34.9 %

---





**Table 25. Dyebath Exhaustion for Sulfogene Tan 2RCF  
at 190 Degrees, Fahrenheit, for Sixty Minutes**

---

Weight of Yarn (Package Number 5)	426.2 grams
Weight of Dye	42.62 grams
Weight of Sodium Carbonate	42.62 grams
Weight of Crystalline Sodium Sulfide	170.48 grams
Weight of Sodium Chloride	85.24 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	3.044 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	69.5 %
Corresponding Concentration	1.945 gms/L
Percentage Exhaustion of Dyebath	36.1 %
Weight of Yarn (Package Number 17)	446.1 grams
Weight of Dye	44.61 grams
Weight of Sodium Carbonate	44.61 grams
Weight of Crystalline Sodium Sulfide	178.44 grams
Weight of Sodium Chloride	89.22 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	3.186 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	67.5 %
Corresponding Concentration	2.113 gms/L
Percentage Exhaustion of Dyebath	34.3 %
Average Exhaustion of Dyebaths	35.2 %

---



**Table 26. Dyebath Exhaustion for Sulfogene Tan 2RCF  
at High Temperature**

**Time of Dyeing: Forty minutes (including 15 minutes  
at 250 degrees, F.)**

---

Weight of Yarn (Package Number 6)	421.8 grams
Weight of Dye	42.18 grams
Weight of Sodium Carbonate	42.18 grams
Weight of Crystalline Sodium Sulfide	168.72 grams
Weight of Sodium Chloride	84.36 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	3.013 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	74.0 %
Corresponding Concentration	1.603 gms/L
Percentage Exhaustion of Dyebath	46.8 %
Weight of Yarn (Package Number 18)	432.0 grams
Weight of Dye	43.2 grams
Weight of Sodium Carbonate	43.2 grams
Weight of Crystalline Sodium Sulfide	172.8 grams
Weight of Sodium Chloride	86.4 grams
Volume of Dyebath	14.0 liters
Initial Concentration of Dyebath	3.056 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	73.6 %
Corresponding Concentration	1.650 gms/L
Percentage Exhaustion of Dyebath	46.5 %
Average Exhaustion of Dyebaths	46.65 %

---

TABLE 10. Monthly Discharge by Station for 1960

Units: cubic feet per second (cfs) for stations 1-10; million cfs for stations 11-15

Station	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Jan	1.2	1.5	1.8	2.1	2.4	2.7	3.0	3.3	3.6	3.9	4.2	4.5	4.8	5.1	5.4
Feb	1.3	1.6	1.9	2.2	2.5	2.8	3.1	3.4	3.7	4.0	4.3	4.6	4.9	5.2	5.5
Mar	1.4	1.7	2.0	2.3	2.6	2.9	3.2	3.5	3.8	4.1	4.4	4.7	5.0	5.3	5.6
Apr	1.5	1.8	2.1	2.4	2.7	3.0	3.3	3.6	3.9	4.2	4.5	4.8	5.1	5.4	5.7
May	1.6	1.9	2.2	2.5	2.8	3.1	3.4	3.7	4.0	4.3	4.6	4.9	5.2	5.5	5.8
Jun	1.7	2.0	2.3	2.6	2.9	3.2	3.5	3.8	4.1	4.4	4.7	5.0	5.3	5.6	5.9
Jul	1.8	2.1	2.4	2.7	3.0	3.3	3.6	3.9	4.2	4.5	4.8	5.1	5.4	5.7	6.0
Aug	1.9	2.2	2.5	2.8	3.1	3.4	3.7	4.0	4.3	4.6	4.9	5.2	5.5	5.8	6.1
Sep	2.0	2.3	2.6	2.9	3.2	3.5	3.8	4.1	4.4	4.7	5.0	5.3	5.6	5.9	6.2
Oct	2.1	2.4	2.7	3.0	3.3	3.6	3.9	4.2	4.5	4.8	5.1	5.4	5.7	6.0	6.3
Nov	2.2	2.5	2.8	3.1	3.4	3.7	4.0	4.3	4.6	4.9	5.2	5.5	5.8	6.1	6.4
Dec	2.3	2.6	2.9	3.2	3.5	3.8	4.1	4.4	4.7	5.0	5.3	5.6	5.9	6.2	6.5
Year	2.4	2.7	3.0	3.3	3.6	3.9	4.2	4.5	4.8	5.1	5.4	5.7	6.0	6.3	6.6
1960	2.5	2.8	3.1	3.4	3.7	4.0	4.3	4.6	4.9	5.2	5.5	5.8	6.1	6.4	6.7
1961	2.6	2.9	3.2	3.5	3.8	4.1	4.4	4.7	5.0	5.3	5.6	5.9	6.2	6.5	6.8
1962	2.7	3.0	3.3	3.6	3.9	4.2	4.5	4.8	5.1	5.4	5.7	6.0	6.3	6.6	6.9
1963	2.8	3.1	3.4	3.7	4.0	4.3	4.6	4.9	5.2	5.5	5.8	6.1	6.4	6.7	7.0
1964	2.9	3.2	3.5	3.8	4.1	4.4	4.7	5.0	5.3	5.6	5.9	6.2	6.5	6.8	7.1
1965	3.0	3.3	3.6	3.9	4.2	4.5	4.8	5.1	5.4	5.7	6.0	6.3	6.6	6.9	7.2
1966	3.1	3.4	3.7	4.0	4.3	4.6	4.9	5.2	5.5	5.8	6.1	6.4	6.7	7.0	7.3
1967	3.2	3.5	3.8	4.1	4.4	4.7	5.0	5.3	5.6	5.9	6.2	6.5	6.8	7.1	7.4
1968	3.3	3.6	3.9	4.2	4.5	4.8	5.1	5.4	5.7	6.0	6.3	6.6	6.9	7.2	7.5
1969	3.4	3.7	4.0	4.3	4.6	4.9	5.2	5.5	5.8	6.1	6.4	6.7	7.0	7.3	7.6
1970	3.5	3.8	4.1	4.4	4.7	5.0	5.3	5.6	5.9	6.2	6.5	6.8	7.1	7.4	7.7
1971	3.6	3.9	4.2	4.5	4.8	5.1	5.4	5.7	6.0	6.3	6.6	6.9	7.2	7.5	7.8
1972	3.7	4.0	4.3	4.6	4.9	5.2	5.5	5.8	6.1	6.4	6.7	7.0	7.3	7.6	7.9
1973	3.8	4.1	4.4	4.7	5.0	5.3	5.6	5.9	6.2	6.5	6.8	7.1	7.4	7.7	8.0
1974	3.9	4.2	4.5	4.8	5.1	5.4	5.7	6.0	6.3	6.6	6.9	7.2	7.5	7.8	8.1
1975	4.0	4.3	4.6	4.9	5.2	5.5	5.8	6.1	6.4	6.7	7.0	7.3	7.6	7.9	8.2
1976	4.1	4.4	4.7	5.0	5.3	5.6	5.9	6.2	6.5	6.8	7.1	7.4	7.7	8.0	8.3
1977	4.2	4.5	4.8	5.1	5.4	5.7	6.0	6.3	6.6	6.9	7.2	7.5	7.8	8.1	8.4
1978	4.3	4.6	4.9	5.2	5.5	5.8	6.1	6.4	6.7	7.0	7.3	7.6	7.9	8.2	8.5
1979	4.4	4.7	5.0	5.3	5.6	5.9	6.2	6.5	6.8	7.1	7.4	7.7	8.0	8.3	8.6
1980	4.5	4.8	5.1	5.4	5.7	6.0	6.3	6.6	6.9	7.2	7.5	7.8	8.1	8.4	8.7
1981	4.6	4.9	5.2	5.5	5.8	6.1	6.4	6.7	7.0	7.3	7.6	7.9	8.2	8.5	8.8
1982	4.7	5.0	5.3	5.6	5.9	6.2	6.5	6.8	7.1	7.4	7.7	8.0	8.3	8.6	8.9
1983	4.8	5.1	5.4	5.7	6.0	6.3	6.6	6.9	7.2	7.5	7.8	8.1	8.4	8.7	9.0
1984	4.9	5.2	5.5	5.8	6.1	6.4	6.7	7.0	7.3	7.6	7.9	8.2	8.5	8.8	9.1
1985	5.0	5.3	5.6	5.9	6.2	6.5	6.8	7.1	7.4	7.7	8.0	8.3	8.6	8.9	9.2
1986	5.1	5.4	5.7	6.0	6.3	6.6	6.9	7.2	7.5	7.8	8.1	8.4	8.7	9.0	9.3
1987	5.2	5.5	5.8	6.1	6.4	6.7	7.0	7.3	7.6	7.9	8.2	8.5	8.8	9.1	9.4
1988	5.3	5.6	5.9	6.2	6.5	6.8	7.1	7.4	7.7	8.0	8.3	8.6	8.9	9.2	9.5
1989	5.4	5.7	6.0	6.3	6.6	6.9	7.2	7.5	7.8	8.1	8.4	8.7	9.0	9.3	9.6
1990	5.5	5.8	6.1	6.4	6.7	7.0	7.3	7.6	7.9	8.2	8.5	8.8	9.1	9.4	9.7
1991	5.6	5.9	6.2	6.5	6.8	7.1	7.4	7.7	8.0	8.3	8.6	8.9	9.2	9.5	9.8
1992	5.7	6.0	6.3	6.6	6.9	7.2	7.5	7.8	8.1	8.4	8.7	9.0	9.3	9.6	9.9
1993	5.8	6.1	6.4	6.7	7.0	7.3	7.6	7.9	8.2	8.5	8.8	9.1	9.4	9.7	10.0
1994	5.9	6.2	6.5	6.8	7.1	7.4	7.7	8.0	8.3	8.6	8.9	9.2	9.5	9.8	10.1
1995	6.0	6.3	6.6	6.9	7.2	7.5	7.8	8.1	8.4	8.7	9.0	9.3	9.6	9.9	10.2
1996	6.1	6.4	6.7	7.0	7.3	7.6	7.9	8.2	8.5	8.8	9.1	9.4	9.7	10.0	10.3
1997	6.2	6.5	6.8	7.1	7.4	7.7	8.0	8.3	8.6	8.9	9.2	9.5	9.8	10.1	10.4
1998	6.3	6.6	6.9	7.2	7.5	7.8	8.1	8.4	8.7	9.0	9.3	9.6	9.9	10.2	10.5
1999	6.4	6.7	7.0	7.3	7.6	7.9	8.2	8.5	8.8	9.1	9.4	9.7	10.0	10.3	10.6
2000	6.5	6.8	7.1	7.4	7.7	8.0	8.3	8.6	8.9	9.2	9.5	9.8	10.1	10.4	10.7
2001	6.6	6.9	7.2	7.5	7.8	8.1	8.4	8.7	9.0	9.3	9.6	9.9	10.2	10.5	10.8
2002	6.7	7.0	7.3	7.6	7.9	8.2	8.5	8.8	9.1	9.4	9.7	10.0	10.3	10.6	10.9
2003	6.8	7.1	7.4	7.7	8.0	8.3	8.6	8.9	9.2	9.5	9.8	10.1	10.4	10.7	11.0
2004	6.9	7.2	7.5	7.8	8.1	8.4	8.7	9.0	9.3	9.6	9.9	10.2	10.5	10.8	11.1
2005	7.0	7.3	7.6	7.9	8.2	8.5	8.8	9.1	9.4	9.7	10.0	10.3	10.6	10.9	11.2
2006	7.1	7.4	7.7	8.0	8.3	8.6	8.9	9.2	9.5	9.8	10.1	10.4	10.7	11.0	11.3
2007	7.2	7.5	7.8	8.1	8.4	8.7	9.0	9.3	9.6	9.9	10.2	10.5	10.8	11.1	11.4
2008	7.3	7.6	7.9	8.2	8.5	8.8	9.1	9.4	9.7	10.0	10.3	10.6	10.9	11.2	11.5
2009	7.4	7.7	8.0	8.3	8.6	8.9	9.2	9.5	9.8	10.1	10.4	10.7	11.0	11.3	11.6
2010	7.5	7.8	8.1	8.4	8.7	9.0	9.3	9.6	9.9	10.2	10.5	10.8	11.1	11.4	11.7
2011	7.6	7.9	8.2	8.5	8.8	9.1	9.4	9.7	10.0	10.3	10.6	10.9	11.2	11.5	11.8
2012	7.7	8.0	8.3	8.6	8.9	9.2	9.5	9.8	10.1	10.4	10.7	11.0	11.3	11.6	11.9
2013	7.8	8.1	8.4	8.7	9.0	9.3	9.6	9.9	10.2	10.5	10.8	11.1	11.4	11.7	12.0
2014	7.9	8.2	8.5	8.8	9.1	9.4	9.7	10.0	10.3	10.6	10.9	11.2	11.5	11.8	12.1
2015	8.0	8.3	8.6	8.9	9.2	9.5	9.8	10.1	10.4	10.7	11.0	11.3	11.6	11.9	12.2
2016	8.1	8.4	8.7	9.0	9.3	9.6	9.9	10.2	10.5	10.8	11.1	11.4	11.7	12.0	12.3
2017	8.2	8.5	8.8	9.1	9.4	9.7	10.0	10.3	10.6	10.9	11.2	11.5	11.8	12.1	12.4
2018	8.3	8.6	8.9	9.2	9.5	9.8	10.1	10.4	10.7	11.0	11.3	11.6	11.9	12.2	12.5
2019	8.4	8.7	9.0	9.3	9.6	9.9	10.2	10.5	10.8	11.1	11.4	11.7	12.0	12.3	12.6
2020	8.5	8.8	9.1	9.4	9.7	10.0	10.3	10.6	10.9	11.2	11.5	11.8	12.1	12.4	12.7
2021	8.6	8.9	9.2	9.5	9.8	10.1	10.4	10.7	11.0	11.3	11.6	11.9	12.2	12.5	12.8
2022	8.7	9.0	9.3	9.6	9.9	10.2	10.5	10.8	11.1	11.4	11.7	12.0	12.3	12.6	12.9
2023	8.8	9.1	9.4	9.7	10.0	10.3	10.6	10.9	11.2	11.5	11.8	12.1	12.4	12.7	13.0
2024	8.9	9.2	9.5	9.8	10.1	10.4	10.7	11.0	11.3	11.6	11.9	12.2	12.5	12.8	13.1
2025	9.0	9.3	9.6	9.9	10.2	10.5	10.8	11.1	11.4	11.7	12.0	12.3	12.6	12.9	13.2
2026	9.1	9.4	9.7	10.0	10.3	10.6	10.9	11.2	11.5	11.8	12.1	12.4	12.7	13.0	13.3
2027	9.2	9.5	9.8	10.1	10.4	10.7	11.0	11.3	11.6	11.9	12.2	12.5	12.8	13.1	13.4
2028	9.3	9.6	9.9	10.2	10.5	10.8	11.1	11.4	11.7	12.0	12.3	12.6	12.9	13.2	13.5
2029	9.4	9.7	10.0	10.3	10.6	10.9	11.2	11.5	11.8	12.1	12.4	12.7	13.0	13.3	13.6
2030	9.5	9.8	10.1	10.4	10.7	11.0	11.3	11.6	11.9	12.2	12.5	12.8	13.1	13.4	13.7
2031	9.6	9.9	10.2	10.5	10.8	11.1	11.4	11.7	12.0	12.3	12.6	12.9	13.2	13.5	13.8
2032	9.7	10.0	10.3	10.6	10.9	11.2	11.5	11.8	12.1	12.4	12.7	13.0	13.3	13.6	13.9
2033	9.8	10.1	10.4	10.7	11.0	11.3	11.6	11.9	12.2	12.5	12.8	13.1	13.4	13.7	14.0
2034	9.9	10.2	10.5	10.8	11.1	11.4	11.7	12.0	12.3						



**Table 27. Dyebath Exhaustion for Sulfogene Direct Blue BRCF Conc. 200%, at 190 Degrees, Fahrenheit, for Sixty Minutes**

---

Weight of Yarn (Package Number 7)	405.0	grams
Weight of Dye	40.5	grams
Weight of Sodium Carbonate	40.5	grams
Weight of Crystalline Sodium Sulfide	121.5	grams
Weight of Sodium Chloride	81.0	grams
Volume of Dyebath	14.0	liters
Initial Concentration of Dyebath	2.8929	gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	63.4	%
Corresponding Concentration	2.0375	gms/L
Percentage Exhaustion of Dyebath	29.6	%
Weight of Yarn (Package Number 19)	407.9	grams
Weight of Dye	40.79	grams
Weight of Sodium Carbonate	40.79	grams
Weight of Crystalline Sodium Sulfide	122.37	grams
Weight of Sodium Chloride	81.58	grams
Volume of Dyebath	14.00	liters
Initial Concentration of Dyebath	2.9138	gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	65.0	%
Corresponding Concentration	1.9275	gms/L
Percentage Exhaustion of Dyebath	33.8	%
Average Exhaustion of Dyebaths	31.7	%

---



**Table 28. Dyebath Exhaustion for Sulfogene Direct Blue  
BRCP Conc. 200% at High Temperature**

---

Weight of Yarn (Package Number 8)	419.8 grams
Weight of Dye	41.98 grams
Weight of Sodium Carbonate	41.88 grams
Weight of Crystalline Sodium Sulfide	125.94 grams
Weight of Sodium Chloride	83.96 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	2.9986 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	64.9 %
Corresponding Concentration	1.9275 gms/L
Percentage Exhaustion of Dyebath	35.7 %
Weight of Yarn (Package Number 20)	458.3 grams
Weight of Dye	45.83 grams
Weight of Sodium Carbonate	45.83 grams
Weight of Crystalline Sodium Sulfide	137.49 grams
Weight of Sodium Chloride	91.66 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	3.2736 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	62.4 %
Corresponding Concentration	2.1123 gms/L
Percentage Exhaustion of Dyebath	35.5 %
Average Exhaustion of Dyebaths	35.6 %

---





**Table 29. Dyebath Exhaustion for Sulfogene Brilliant Green GCF Extra Conc. 150% at 190 Degrees, Fahrenheit, for Sixty Minutes**

---

Weight of Yarn (Package Number 9)	434.9 grams
Weight of Dye	43.49 grams
Weight of Sodium Carbonate	43.49 grams
Weight of Crystalline Sodium Sulfide	43.49 grams
Weight of Sodium Chloride	86.98 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	3.1064 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	80.0 %
Corresponding Concentration	1.7125 gms/L
Percentage Exhaustion of Dyebath	44.9 %
Weight of Yarn (Package Number 21)	393.5 grams
Weight of Dye	39.35 grams
Weight of Sodium Carbonate	39.35 grams
Weight of Crystalline Sodium Sulfide	39.35 grams
Weight of Sodium Chloride	78.70 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	2.8107 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	81.0 %
Corresponding Concentration	1.6125 gms/L
Percentage Exhaustion of Dyebath	42.6 %
Average Exhaustion of Dyebaths	43.8 %

---



**Table 30. Dyebath Exhaustion for Sulfogene Brilliant  
Green GCF Extra Conc. 150% at High Temperature**

---

Weight of Yarn (Package Number 10)	441.3 grams
Weight of Dye	44.13 grams
Weight of Sodium Carbonate	44.13 grams
Weight of Crystalline Sodium Sulfide	44.13 grams
Weight of Sodium Chloride	88.26 grams
Volume of Dyebath	14.00 liters
Initial Concentration of Dyebath	3.1521 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	79.7 %
Corresponding Concentration	1.7375 gms/L
Percentage Exhaustion of Dyebath	44.9 %
Weight of Yarn (Package Number 22)	427.0 grams
Weight of Dye	42.7 grams
Weight of Sodium Carbonate	42.7 grams
Weight of Crystalline Sodium Sulfide	42.7 grams
Weight of Sodium Chloride	42.7 grams
Volume of Dyebath	14.0 liters
Initial Concentration of Dyebath	3.0500 gms/L
Transmission of Light of Exhausted Dyebath (Diluted to one one-hundredth concentration)	81.0 %
Corresponding Concentration	1.6125 gms/L
Percentage Exhaustion of Dyebath	47.1 %
Average Exhaustion of Dyebaths	46.0 %

---





Table 31. Comparison of Residual Strengths of Dyed Yarns

Sulfogene Dyes	Normal Temperature	High Temperature
Carbon BCF Grains	Package #1	Package #2
Break-Strength Before Dyeing	1.21 lbs.	1.11 lbs.
Break-Strength After Dyeing	1.06	1.12
Change in Strength	0.15 lb. loss	0.01 lb. gain
Navy Blue 4RCF Supra	Package #3	Package #4
Break-Strength Before Dyeing	1.04 lbs.	1.10 lbs.
Break-Strength After Dyeing	1.11	1.09
Change in Strength	0.07 lb. gain	0.01 lb. loss
Tan 2RCF	Package #5	Package #6
Break-Strength Before Dyeing	1.50 lbs.	0.97 lbs.
Break-Strength After Dyeing	1.06	1.14
Change in Strength	0.44 lb. loss	0.17 lb. gain
Direct Blue BRCF	Package #7	Package #8
Break-Strength Before Dyeing	1.15 lbs.	1.10 lbs.
Break-Strength After Dyeing	1.13	1.29
Change in Strength	0.02 lb. loss	0.19 lb. gain
Brilliant Green GCF	Package #9	Package #10
Break-Strength Before Dyeing	1.06 lbs.	1.11 lbs.
Break-Strength After Dyeing	1.23	1.13
Change in Strength	0.17 lb. gain	0.02 lb. gain



**BIBLIOGRAPHY**





## LITERATURE CITED

1. Zimmerman, Charles L., "The Application of Dyes to Textile Fibers at High Temperatures", American Dyestuff Reporter, 42 (September 14, 1953), pp. 627-634.
2. Venkataraman, K., The Chemistry of Synthetic Dyes, Vol. I. New York: Academic Press, Inc., Publishers, 1952, p. 63.
3. Ibid., p. 274.
4. Drijvers, Ir. L., "Dyeing of Textile Fibers at High Temperatures (Above 100° C.)", Textielwezen, (August, 1952), pp. 18-37.
5. Philadelphia Section, American Association of Textile Chemists and Colourists, "Some Phases of High Temperature Dyeing", American Dyestuff Reporter, 1 (January 10, 1949), pp. 9-32.
6. A.S.T.M. Committee D-13 on Textile Materials, A.S.T.M. Standards on Textile Materials. Philadelphia: American Society for Testing Materials, 1952, pp. 337-343.
7. Young, Ernest H., An Evaluation of the Use of High Temperature Procedures for Applying Direct Dyes to Cotton Yarn, A Thesis. Atlanta: Georgia Institute of Technology, 1953, p. 31.
8. Vickerstaff, Thomas, The Physical Chemistry of Dyeing. New York: Interscience Publishers, Inc., 1950, pp. 29-43.
9. 1953 Technical Manual and Yearbook of the American Association of Textile Chemists and Colorists, Volume XXIX. New York: Howes Publishing Co., Inc., 1953, pp. 103-111.
10. Ibid., pp. 91-92.

## OTHER REFERENCES

Beach, Franklin, The Dyeing of Cotton Fabrics, Third Edition, Revised and Enlarged by A. J. Hall. New York: D. Van Nostrand Company, 1927, pp. 89-91, 191-193.





Birchall, H., "Developing and Applying Sulfur Dyes to Loose Cotton and Cotton Yarns", *Journal of the Society of Dyers and Colourists*, 67 (1951), pp. 495-501.

Butterworth, K., "The Dyeing of Viscose Rayon at High Temperatures", *Journal of the Society of Dyers and Colourists*, 69 (1953), pp. 362-370.

Diserens, Louis, *The Chemical Technology of Dyeing and Printing - Vat, Sulfur, Indigosol, Azo and Chrome Dyestuffs and Their Auxiliaries*, Vol. I. Translated and Revised from Second German Edition by Paul Wengraf and Herman P. Baumann. New York: Reinhold Publishing Corp., 1948, pp. 161-174.

Eaton, J. C., C. H. Giles, and Manfred Gordon, "Quantitative Relation Between Depth of Dyeing and Light Fastness", *Journal of the Society of Dyers and Colourists*, 68 (1952), pp. 394-396.

Hartung, K., "Dyeing at High Temperatures (Over 100°)", *Textil-Praxis*, 6 (1951), pp. 204-6.

Horsfall, R. S., and L. G. Lowrie, *The Dyeing of Textile Fibers*, Second Edition Reprinted. London: Chapman & Hall, Ltd., 1949, pp. 98-102.

Jackson, J. H. E., and H. A. Turner, "The Desorption of a Direct Cotton Dye from Cellulose Fibers", *Journal of the Society of Dyers and Colourists*, 68 (1952), pp. 345-352.

Millson, H. E., and L. H. Turl, "Microscopic Dyeing Phenomena; Studies with the Micro-dyeoscope", *Textile Research Journal*, 21, (1951), pp. 658-702.

Robinson, R. D., and C. L. Zimmerman, "Some Practical Aspects of High Temperature Dyeing", *American Dyestuff Reporter*, 39 (April 17, 1950), pp. 250-255.

Royer, G. L., and others, "Dyeing Studies at Elevated Temperatures Between 200° F. and 300° F.", *Textile Research Journal*, 18 (October, 1948), pp. 598-614.

Wahl, Andre, *The Manufacture of Organic Dyestuffs*. Authorized Translation with Additions, from the French by F. W. Atack. London: G. Bell and Sons, Ltd., 1919, p. 304.





Whittaker, C. M., Dyeing with Coal-tar Dyestuffs. Second Edition. London: Bailliere, Tindall and Cox, 1926, pp. 99-120.

Zimmerman, C. M., "Application of Dyes to Textile Fibers at High Temperatures", American Dyestuff Reporter, 42, Proceedings of the American Association of Textile Chemists and Colourists, 42 (1953), pp. 627-634.

"No Hocus Pocus at Ankokas", staff article, Textile Industries, 118 (April, 1954), pp. 164-167.













OCT 27

DISPLAY

Thesis  
M96

Murauskas

25082

Application of sulfur  
dyes on cotton at high  
temperatures.

OCT 27

DISPLAY

2

25082

Thesis  
M96

Murauskas

Application of sulfur dyes on  
cotton at high temperature.

thesM96

Application of sulphur dyes on cotton at



3 2768 000 99350 5

DUDLEY KNOX LIBRARY